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TECHNOLOGY IN ACTION

LIQUEFACTION OF NOVA SCOTIA COALS
PHASE VII

STUDIES ON INTEGRATED TWO-STAGE THERMAL/CATALYTIC LIQUEFACTION

FINAL REPORT

by

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SUMMARY

Phase VII of a research program on the direct liquefaction of Nova Scotia bituminous coals has been completed. During this phase of work bench-scale studies were carried out on integrated two-stage thermal/catalytic liquefaction of Sydney area coals. Two conventional hydroprocessing catalysts, Katalco 550 and Amocat 1C, were tested in the second-stage reactor. The former is a nickel-tungsten catalyst with a unimodal pore size distribution. The latter is a nickel-molybdenum bimodal catalyst.

A total of eighteen liquefaction experiments were conducted in a once-through process mode using hydrogenated anthracene oil, 9%H, as reaction solvent. Experiments were carried out with Lingan Harbour, Prince Hub and Donkin Harbour seam coals to investigate the effects of operating conditions on coal conversion and raw product distribution. Illinois No. 6 coal was tested as a benchmark feedstock.

High coal conversions, 92 - 95 wt.%, were achieved with both Katalco 550 and Amocat 1C. Experimental results have shown that thermal dissolver temperature over the range of 430 to 460°C and catalytic reactor temperature over the range of 400 to 435°C significantly affect product composition and hydrogen consumption. The highest oil yields produced with Lingan Harbour, Donkin Harbour and Prince Hub seam coals were 51, 54 and 56 wt.% respectively, compared to 59 wt.% with Illinois No. 6 coal. Amocat 1C exhibited superior activity for the conversion of coal to pentane soluble oil. The superior activity of this nickel-molybdenum catalyst is attributed to the bimodal pore size distribution on the basis of earlier work with a unimodal nickel-molybdenum catalyst, Katalco 504-K.

On completion of the exploratory tests, a six-day recycle liquefaction run was conducted with Amocat 1C using Prince Hub seam coal. Conversion averaged 91 wt.% over the last twelve-hour operating period. The net C_4^+ liquid yield was 67 wt.% based on maf coal plus hydrogen added. High yields of light and middle distillates were achieved in this test. The yields of naphtha and light gas oil were 44 and 38 wt.% respectively.

The next phase of work will concentrate on process optimization, coal reactivity and catalyst evaluation studies. Additional test work is planned on recycle liquefaction of Prince Hub seam coal. On completion of the recycle liquefaction work, the first-stage thermal dissolver will be converted to a catalytic dissolver. Following this, a test program will be undertaken on catalytic two-stage liquefaction of Sydney area coals.

INTRODUCTION

The Organization of Petroleum Exporting Countries was established in 1960 to gain control over oil prices from the multinational oil companies. By 1973, the economic well-being of industrial nations that were dependent wholly or in part on foreign sources of crude oil was threatened by energy crises, feedstock shortages and soaring fuel costs. The unpredictability of the international oil market influenced Western governments to intensify conservation efforts and expand their domestic energy base. As a consequence of the energy strategies formulated by Western nations, considerable attention has been focused on the production of liquid fuels from coal in recent years.

Historically, two routes have been used to produce hydrocarbon liquids from coal - a direct or hydroliquefaction route and an indirect or synthesis route. During the past fifteen years, significant advances have been made relating to the production of synthetic liquid fuels via coal liquefaction. Within the past decade, large-scale pilot plants have been built in the United States and Germany to determine the economic feasibility of new direct liquefaction processes, e.g., Exxon Donor Solvent, H-Coal and Ruhrkohle/Veba.

Since 1982 the international oil market has stabilized and the world price of crude oil has declined substantially. This has resulted in the cancellation or slow-down of most large-scale synfuels projects in North America. Nevertheless, a modest level of research is being maintained to pursue second generation processes. In other parts of

the world industrialized nations including England, Germany and Japan are still actively conducting programs to further develop coal liquefaction technology.

In Canada, our way of life is highly dependent on petroleum liquids. Since our existing crude supplies are not expected to meet our future liquid fuel demands, it seems that we will have to rely on synthetic liquid fuels to some degree in the next few decades. Thus, should it be economically attractive or essential, abundant coal reserves are available in Canada for conversion to synthetic liquid fuels. Because long lead times are involved in process evaluation, detailed design and plant construction, it is essential to understand the basic concepts of the promising liquefaction processes and verify their potential yield structure as they apply to Canadian coals.

The in-situ coal resources of Nova Scotia occur in five main fields: Sydney, Mabou, Port Hood, Pictou and Springhill. The largest resources, over 2 billion metric tons, occur in the submarine area of the Sydney coalfield. The coal-bearing section of the Sydney basin contains eleven major seams ranging from 1 - 4.5 m thick. Historically, the two most productive seams have been the Harbour and Phalen seams.

At present, the principal coal producer in the province is the Cape Breton Development Corporation with two operating mines, Lingan and Prince, and a third under development at Donkin. All three mines are located in the Sydney area. The Harbour seam is currently being

worked at Lingan whereas Hub seam coal is being produced from the Prince mine. Both coals are classified as high volatile A bituminous.

Process studies relating to the direct liquefaction of Nova Scotia high volatile bituminous coals were initiated at the Nova Scotia Research Foundation Corporation (NSRFC) in 1978. Initially, efforts were concentrated on a low-severity extractive coking process (1,2,3). From 1980 to 1984, experimental work was conducted on medium to high pressure donor extraction (4,5) and single-stage catalytic hydrogenation (6). Yield data has been developed over a wide range of operating conditions using bench-scale batch and continuous-flow hydroprocessing units. The product slate of interest is liquid transportation fuels.

Test results have shown that single-stage catalytic hydrogenation produces higher oil yields than donor solvent extraction. Furthermore, it has been shown that considerable control can be exercised over product distribution by varying process severity. Once-through experiments with Lingan Harbour seam coal and commercial nickel-molybdenum hydrotreating catalyst over medium to high severity operating conditions produced high coal conversions, 90 - 92%, while oil yields ranged from 30 - 47%.

Direct catalytic coal liquefaction processes, such as H-Coal, are single-stage processes. The catalyst serves several functions and the severe operating conditions required for one function might not be needed for other functions. Recent process developments in the United

States are centered on liquefying coal in two separate reaction zones. Two-stage processing offers the versatility of controlling coal dissolution and primary product upgrading under different operating conditions.

During the first half of 1985 our bench-scale coal liquefaction unit was modified and expanded for integrated two-stage operations. The closely coupled two reactor system was built because two-stage processing offers the potential of higher distillate yields and improved hydrogen utilization. The two-stage unit has been operated in a thermal/catalytic mode since mid-1985. Initially, experimental work was concentrated on Lingan Harbour seam coal using nickel-molybdenum and nickel-tungsten unimodal micropore catalysts in the second-stage reactor. Test results have shown that coal space velocity, total pressure and catalytic reactor temperature significantly affect product composition and hydrogen consumption (7).

In 1986 a total of fifteen experiments were conducted in a once-through mode to further investigate the reactivity of Sydney area coals under two-stage thermal/catalytic liquefaction conditions. Three additional runs were made with Illinois No. 6 coal for comparative purposes. Six of the eighteen experiments were carried out with Katalco 550 nickel-tungsten unimodal catalyst, the remainder with Amocat 1C nickel-molybdenum bimodal catalyst. After completion of the eighteen exploratory tests, a six-day run with liquid recycle was undertaken using Prince Hub seam coal. The results of these tests are presented herein.

TASK 1 - EQUIPMENT MAINTENANCE

Actual work on Phase VII began in December of 1985. Activities under Task 1 included the installation of a new hydrogen compressor, inspection of equipment and general maintenance associated with the hydrogen compression facility, the fixed-bed catalytic hydrotreater and the two-stage coal liquefaction unit.

The single-stage diaphragm compressor purchased during Phase VI was installed in January, 1986. The unit was acquired to replace our ageing hydrogen compressor and to provide increased capacity for longer term coal liquefaction tests. The design capacity of the new 7.5 horsepower unit is 114 L/min from 3.5 MPa to 34.5 MPa. Installation and commissioning of the unit went smoothly. The new compressor performed very well throughout this phase of work.

The fixed-bed catalytic hydrotreater has been described in an earlier report (3). On turnaround, the high pressure liquid metering pump was given a general overhaul and new packing rings were installed in the pump body. Heating tapes and insulation on the feed vessel and liquid transfer lines between the feed vessel and the reactor inlet were replaced. In addition, high pressure valves on both feed and product vessels were replaced.

A complete description of the two-stage coal liquefaction unit is presented elsewhere (7). The period allocated for inspection and maintenance of the unit was approximately two weeks. Routine maintenance was performed on the low pressure feed circuit, the high

pressure slurry metering pump and the catalytic reactor internals in a very timely manner. Furthermore, several high pressure block valves and product let-down valves were rebuilt or replaced during the two week period. Completion of Task 1 slipped by about four weeks, however, due to unexpected problems experienced with both reactors and the automated high pressure let-down system.

Several attempts were required to seal the bolted closure reactors. A satisfactory seal was finally achieved by machining the sealing surfaces in contact with the cover gasket and modifying the procedure for tightening the cover bolts.

During the first trial to reactivate the coal liquefaction unit the high pressure let-down system failed to operate. The problem was traced to a defective amplifier module in the electronics section of the gamma ray ion chamber detector system. A new amplifier board was procured and installed within a week. Following minor adjustments satisfactory operation was achieved.

TASK 2 - PROCUREMENT OF NEW EQUIPMENT

The purpose of this task was to procure new equipment to upgrade and modify our two-stage coal liquefaction unit. Major pieces of equipment purchased were as follows:

(1) Data acquisition and processing system comprised of a Fluke Model 2280B datalogger and Sanyo Model MBC880 personal desktop computer complete with a CRT display monitor and printer.

(2) Equipment rack and data terminal work station to accommodate the data acquisition and processing system mentioned above.

(3) Robinson/Mahoney style internals for the 1.5-liter first-stage reactor. The reactor internals include a catalyst basket and an impeller/baffle system.

(4) Durco single feed metering pump with capacity of 4.4 LPH and maximum discharge pressure of 34.5 MPa.

(5) Autoclave Engineers Inc. digital pressure indicator system complete with two pressure transducers.

(6) Wet test gas flowmeter - a spare unit procured as back-up for the two meters presently in service.

TASK 3 - INSTALLATION OF NEW EQUIPMENT

Activities under Task 3 were related to the installation of equipment described in the previous section. The installation of new equipment was scheduled around other tasks to avoid disruption of bench unit operations.

Our first priority was to install the Durco metering pump. This pump was added to the coal liquefaction unit in parallel with a Hills-McCanna pump of similar design. Thus, either pump can be used to deliver coal-oil slurry to the high pressure reaction zone.

The next priority was to assemble the equipment rack and work station. These components have been installed adjacent to the existing control console. After completion of the mechanical work, additional electrical service was provided to this area to accommodate the data acquisition and processing system.

Progress with regard to installation of the datalogger has been slow. Initially, considerable time was lost because the supplier shipped the wrong cable to interface the datalogger with the printer, which they also supplied. Following this, a few difficulties were encountered interfacing process instrumentation with the datalogger. These problems are currently being addressed. In the meantime, the computer section is fully operational and is being used for data processing associated with bench unit and laboratory operations.

TASK 4 - PREPARATION OF RAW MATERIALS

4.1 Coal Feedstocks

Four coal feedstocks were tested during this phase of the project. The experimental program was primarily focused on three Sydney area coals with less emphasis on Illinois No. 6 from the Burning Star Mine. Prince Hub, Lingan Harbour and Donkin Harbour seam coals were provided courtesy of the Cape Breton Development Corporation. Illinois No. 6 was procured through Cepheus Industries Incorporated, Marion, Illinois. Preparation involved riffling, grinding and screening. After screening, the individual coals were thoroughly mixed and stored in plastic-lined drums with a nitrogen blanket.

Proximate and ultimate analysis of the coals are given in Table 1. Proximate analysis, free-swelling index and forms of sulphur were determined according to standard methods. Total sulphur was measured using a Leco sulphur analyzer. Carbon, hydrogen and nitrogen were determined using a Perkin-Elmer elemental analyzer.

Petrographic analysis of the four coals is given in Table 2. These analyses were carried out by coal petrology experts at the Bedford Institute of Oceanography.

TABLE 1

ANALYSIS OF COALS TESTED

COAL DESCRIPTION:	LINGAN HR-7	DONKIN HR-7	PRINCE HB-7	ILLINOIS No.6
Moisture, Wt. %	1.28	1.65	3.30	3.03
PROXIMATE (Dry Basis) Wt. %				
Ash	3.42	5.01	9.10	11.16
Volatile Matter	37.50	36.64	36.59	38.77
ULTIMATE (Dry Basis) Wt. %				
Carbon	82.03	77.75	70.99	68.43
Hydrogen	5.38	5.19	4.78	4.71
Nitrogen	1.65	1.53	1.53	1.24
Sulphur	1.34	2.73	3.79	3.40
Ash	3.42	5.01	9.10	11.16
Oxygen (by difference)	6.18	7.79	9.81	11.06
SULPHUR FORMS (Dry Basis) Wt. %				
Sulphate	<0.01	0.20	1.10	0.31
Pyritic	0.84	1.26	1.45	1.09
Organic (by difference)	0.50	1.27	1.24	2.00
Total	1.34	2.73	3.79	3.40
FREE SWELLING INDEX	8	7½	1½	1½
HEATING VALUE (kj/kg)	34,002	32,754	29,088	28,223

TABLE 2

PETROGRAPHIC CHARACTERISTICS OF COALS TESTED

COAL DESCRIPTION:	LINGAN HR-7	DONKIN HR-7	PRINCE HB-7	ILLINOIS No.6
MACERAL COMPOSITION (% by Volume)				
Vitrinite	73.4	75.8	74.7	79.2
Exinite	7.6	3.2	4.1	3.8
Inertinite	17.2	16.8	18.0	11.8
Macrinite	(3.6)	(1.4)	(0.8)	(0.6)
Micrinite	(5.6)	(7.8)	(5.4)	(4.6)
Semifusinite	(3.4)	(2.8)	(5.4)	(2.8)
Fusinite	(4.6)	(4.8)	(6.4)	(3.8)
Mineral Matter	1.8	4.2	3.2	5.2
Pyrite	(0.4)	(2.6)	(2.6)	(1.4)
Quartz	(0.2)	(0.4)	--	(1.4)
Shale	(1.2)	(1.2)	(0.4)	(2.4)
Calcite	--	--	(0.2)	--
% R_0 Max.	0.97	0.92	0.78	0.62
V-Type Distribution, %				
V5	--	--	--	32
V6	--	--	6	64
V7	--	2	60	4
V8	12	36	26	--
V9	56	58	2	--
V10	32	4	6	--
V11	--	--	--	--

4.2 Anthracene Oil

This material is a coal-tar distillate which contains ca. 5.8 wt.% hydrogen. It was supplied by Domtar Chemicals Limited (Hamilton, Ontario). Hydrogenated solvents used in the two-stage thermal/catalytic tests were made from three batches of this highly aromatic oil.

The characteristics of the raw oil are presented in Table 3. Carbon and hydrogen were determined using a Perkin-Elmer elemental analyzer. A standard Kjeldahl method was used for the determination of nitrogen. Total sulphur content was measured by x-ray fluorescence. Oxygen was determined by neutron activation.

In this study, the hydrogen aromatic/aliphatic distribution was established by proton nuclear magnetic resonance (H-NMR). A varian EM360L continuous wave spectrometer operating in the Fourier transform mode at 60 MHz, and a Nicolet 360 NB NMR spectrometer operating in the Fourier transform mode at 360 MHz were used to obtain H-NMR spectra. Proton assignments were as follows: aromatic hydrogens (H_{ar}) were found in the region of 9-6 ppm. Benzylic hydrogens, including methyls, occur at 3.5 to 2 ppm (H_{α}). Hydrogens, except methyls, on carbons one or more positions removed from the aromatic rings, occur at 2-1.1 ppm (H_{β}). Aliphatic methyls are found at 1.1-0.4 ppm (H_{γ}). As almost no long aliphatic side chains are believed to be present, the H_{β} protons are predominantly in rings. These assignments are consistent with similar analyses reported in the literature (8,9). Figure 1 is a typical H-NMR spectrum of raw anthracene oil.

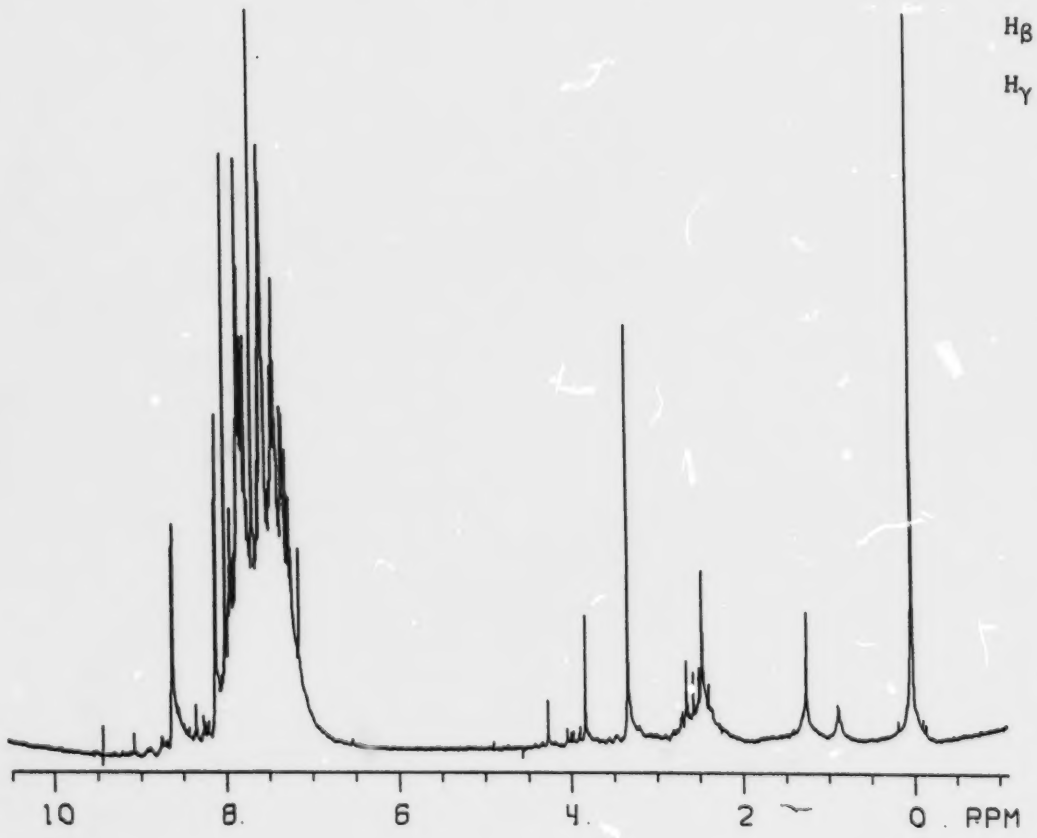
TABLE 3

ANALYSIS OF RAW ANTHRACENE OIL

SAMPLE DESCRIPTION:	BATCH No. 6-13	BATCH No. 6-14	BATCH No. 6-15
Specific Gravity, 60/60°F	1.1325	1.1305	1.1301
Carbon, Wt. %	91.10	90.14	91.64
Hydrogen, Wt. %	5.83	5.90	5.67
Nitrogen, Wt. %	0.91	0.92	0.93
Sulphur, Wt. %	0.62	0.62	0.62
Oxygen, Wt. %	0.71	1.04	1.00
Proton Distribution, %			
H_{ar} (9 - 6 ppm)	84.10	87.02	88.75
H_{α} (3.5 - 2 ppm)	11.00	8.77	7.60
H_{β} (2 - 1.1 ppm)	3.00	1.75	1.21
H_{γ} (1.1 - 0.4 ppm)	2.10	2.46	2.43
Distillation Data °C (ASTM D-1160)			
IBP	229	221	217
10 %	264	262	266
20 %	299	292	289
30 %	321	316	314
40 %	341	335	335
50 %	356	350	353
60 %	370	365	368
70 %	386	380	385
80 %	405	399	405
90 %	442	440	440
95 %	466	467	462
EP	489	484	493

Figure 1 - H-NMR SPECTRA OF RAW ANTHRACENE OIL WITH 5.83 WT. % HYDROGEN

PROTON DISTRIBUTION, %	
H_{ar} (9 - 6 ppm)	84.10
H_{α} (3.5 - 2 ppm)	11.00
H_{β} (2 - 1.1 ppm)	3.00
H_{γ} (1.1 - 0.4 ppm)	2.10



4.3 Catalysts

Two hydroprocessing catalysts, Katalco 550 and Amocat 1C were evaluated in this phase of the two-stage coal liquefaction test program. The former catalyst is alumina-supported $NiO-WO_3$ whereas the latter is alumina-supported $NiO-MoO_3$. Both catalysts were used in extrudate form, 1.6 mm diameter. Katalco 550 has a unimodal pore size distribution and Amocat 1C has a bimodal pore size distribution. Nalcomo 479, an alumina-supported $CoO-MoO_3$ catalyst was used as received in the fixed-bed catalytic hydrotreater. The chemical and physical properties of the three catalysts are given in Table 4.

TABLE 4

CHEMICAL AND PHYSICAL PROPERTIES OF HYDROTREATING CATALYSTS

	KATALCO 550	AMOCAT 1C	NALCOMO 479
MoO_3, Wt. %		16.0	19.0
NiO, Wt. %	5.0	3.0	
CoO, Wt. %			4.4
WO_3, Wt. %	20.0		
Size, cm	0.16	0.16	0.16
Surface Area, M^2/g	230	190	180
Pore Volume, cc/g	0.50	0.85	0.47
Density, g/cc	0.79-0.81	0.67	0.82-0.84
Crush Strength, kg	6.4-7.3	4.4	6.4-7.3
Modal Pore Diameter, A°	100	100/1500	100

TASK 5 - HYDROGENATION OF ANTHRACENE OIL

Hydrogenated solvent used in Task 6 was prepared from raw anthracene oil using a fixed-bed catalytic hydrotreater. The bench-scale unit has been described in an earlier report (3).

A commercial cobalt-molybdenum hydrotreating catalyst, Nalcomo 479, was used to prepare solvent containing 9% hydrogen. A summary of the operating conditions used to produce the hydrogenated oil is given in Table 5. The characteristics of two 9%H oil blends are shown in Table 6. An H-NMR spectrum of the 9%H solvent is presented in Figure 2. Hydrogenated anthracene oil, 7.5%H, spiked with dimethyl disulphide was used to sulphide the Amocat 1C catalyst used in Task 6. The spiked oil was on hand from Phase VI.

Approximately 360 liters of 9%H solvent was produced during this phase of work. The solvent was prepared at the rate of 1.8 liters per 7-hour shift. The catalytic hydrotreater was often operated two shifts per day. Daily production was collected to obtain 20-liter batches. A total of eighteen 20-liter batches were prepared. Analysis of selected batches are presented in Table 7. About 40 liters of this oil was used for catalyst conditioning and post run flush operations. Prior to and during Task 6 test work, 160-liter blends were prepared by mixing eight 20-liter batches. The analysis given in Table 6 is representative of the two 160-liter oil blends.

TABLE 5

REACTION CONDITIONS FOR ANTHRACENE OIL HYDROGENATION

Reactor Temperature, °C.	363-380
Reactor Pressure, MPa	15.51
LHSV, hr^{-1}	0.20
Hydrogen Flow, L/hr	170-270
Product Hydrogen Content, Wt. %	9.0

Catalyst used: Nalcomo 479

TABLE 6

CHARACTERISTICS OF HYDROGENATED ANTHRACENE OIL

SAMPLE DESCRIPTION:	HAO 71	HAO 72
Specific Gravity, 60/60°F	1.0262	1.0268
Carbon, Wt. %	89.73	90.16
Hydrogen, Wt. %	9.01	9.03
Nitrogen, Wt. %	0.10	0.11
Sulphur, Wt. %	0.005	0.007
Oxygen, Wt. %	0.35	0.33
Proton Distribution, %		
H_{ar} (9 - 6 ppm)	26.65	26.07
H_{α} (3.5 - 2 ppm)	26.31	28.74
H_{β} (2 - 1.1 ppm)	36.19	35.22
H_{γ} (1.1 - 0.4 ppm)	10.86	9.99
Distillation Data, °C (ASTM D-1160)		
IBP	185	187
10 %	247	246
20 %	269	268
30 %	288	286
40 %	301	301
50 %	311	310
60 %	322	321
70 %	333	332
80 %	351	349
90 %	374	371
95 %	391	386
EP	446	435

Figure 2 - H-NMR SPECTRA OF HYDROGENATED ANTHRACENE OIL WITH 9.03 WT. % HYDROGEN

PROTON DISTRIBUTION, %	
H_{ar} (9 - 6 ppm)	26.07
H_{α} (3.5 - 2 ppm)	28.74
H_{β} (2 - 1.1 ppm)	35.22
H_{γ} (1.1 - 0.4 ppm)	9.99

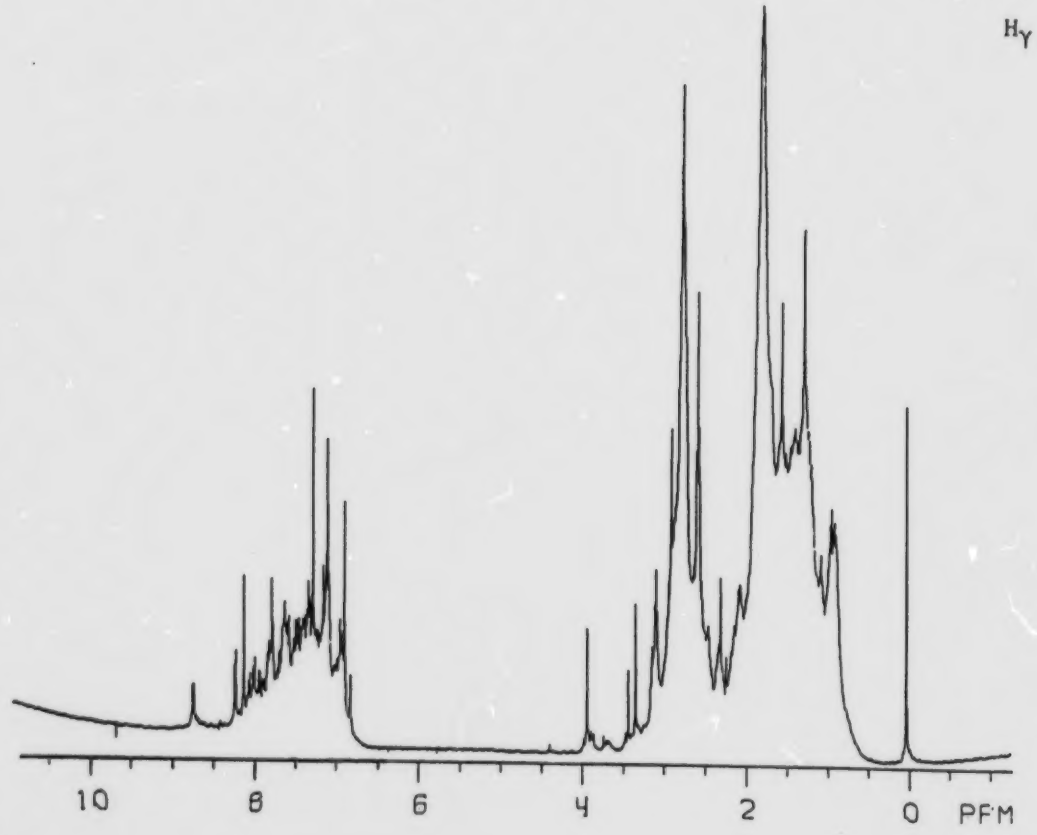


TABLE 7

ANALYSIS OF 20-LITER BATCHES OF 9% H SOLVENT

BATCH NO.	1	2	3	4	5	6	7	8	9
Specific Gravity, 60/60°F	1.0281	1.0278	1.0285	1.0265	1.0255	1.0262	1.0262	1.0238	1.0265
Carbon, Wt. %	89.70	89.87	89.68	89.81	89.98	90.30	89.34	90.15	88.97
Hydrogen, Wt. %	8.96	9.04	8.99	8.95	9.00	9.05	9.03	9.13	8.92
Nitrogen, Wt. %	0.12	0.12	0.10	0.09	0.09	0.09	0.09	0.09	0.10
Sulphur, Wt. %	0.009	0.009	0.005	0.005	0.006	0.006	0.003	0.005	0.005
Proton Distribution, %									
H_{ar} (9 - 6 ppm)	27.75		26.88		27.43			23.38	24.31
H_{α} (3.5 - 2 ppm)	27.17		28.22		27.39			27.40	29.91
H_{β} (2 - 1.1 ppm)	36.42		35.12		36.09			37.75	35.98
H_{γ} (1.1 - 0.4 ppm)	8.67		9.79		9.10			11.47	9.84
Distillation Data, °C (ASTM D-86)									
IBP	188	166	183	162	168	170	159	168	164
5 %	245	236	242	236	236	235	234	233	234
10 %	265	248	260	248	257	252	253	252	252
20 %	280	276	278	272	276	273	273	274	272
30 %	291	290	293	286	290	289	288	288	288
40 %	304	303	307	300	304	303	300	300	301
50 %	315	315	318	314	315	314	313	315	312
60 %	326	325	328	325	326	325	323	324	322
70 %	338	338	340	337	339	338	335	335	334
80 %	355	354	355	353	354	353	352	352	349
90 %	378	380	384	381	380	381	378	378	378
95%	404	411	420	416	408	410	407	404	401
EP	487	481	515	508	505	502	503	506	499

TABLE 7 (Continued)

ANALYSIS OF 20-LITER BATCHES OF 9% H SOLVENT

BATCH NO.	10	11	12	13	14	15	16	17	18
Specific Gravity, 60/60°F	1.0265	1.0272	1.0265	1.0272	1.0262	1.0252	1.0275	1.0288	1.0275
Carbon, Wt. %	90.16	90.00	90.78	89.35	89.80	90.49	90.01	90.21	89.97
Hydrogen, Wt. %	9.09	9.03	9.15	8.99	9.00	9.26	9.16	9.09	8.97
Nitrogen, Wt. %	0.12	0.12	0.11	0.11	0.11	0.10	0.12	0.12	0.11
Sulphur, Wt. %	0.006	0.005	0.006	0.005	0.004	0.005	0.007	0.006	0.007
Proton Distribution, %									
H_{ar} (9 - 6 ppm)	25.52		25.42		25.15	24.88			27.13
H_{α} (3.5 - 2 ppm)	28.28		28.81		32.16	29.51			28.68
H_{β} (2 - 1.1 ppm)	37.24		35.59		33.33	34.69			34.11
H_{γ} (1.1 - 0.4 ppm)	8.97		10.17		9.36	10.93			10.08
Distillation Data, °C (ASTM D-86)									
IBP	180	185	180	184	178	176	140	144	174
5 %	237	239	236	232	242	236	236	240	239
10 %	251	254	250	250	258	252	253	254	254
20 %	274	275	273	269	275	274	272	274	274
30 %	289	290	288	286	288	289	289	290	290
40 %	301	302	301	299	300	299	301	302	301
50 %	313	315	314	314	314	316	313	314	313
60 %	324	325	324	323	324	323	323	324	323
70 %	336	337	336	335	336	336	336	334	334
80 %	350	353	352	350	352	351	354	352	354
90 %	380	380	380	376	378	379	378	377	386
95 %	399	404	408	397	410	405	400	404	420
EP	491	486	468	462	470	471	512	509	489

TASK 6 - TWO-STAGE THERMAL/CATALYTIC LIQUEFACTION

The purpose of this task was to continue experimental studies on integrated two-stage thermal/catalytic liquefaction applied to Sydney area coals. In the process, coal is solubilized in the thermal first-stage and subsequently upgraded in the catalytic second-stage. By using two separate reaction zones, the thermal dissolution and catalytic upgrading steps can be independently optimized to maximize efficiency and control product distribution and quality.

A schematic diagram of the two-stage bench unit used is shown in Appendix E. The unit has been described previously (7).

6.1 Extension of Operations with Katalco 550 Unimodal Catalyst

Two-stage bench unit operations with Katalco 550 nickel-tungsten catalyst began in Phase VI. This alumina-supported catalyst has a unimodal pore size distribution with an average pore diameter of about 100A°. During Phase VI, test work was concentrated on Lingan Harbour seam coal. In Phase VII, a total of six exploratory runs were conducted with Prince Hub, Donkin Harbour and Illinois No. 6 coals varying thermal dissolver and catalytic reactor temperatures.

The experiments were conducted in a once-through process mode using hydrogenated anthracene oil containing 9%H as reaction solvent. Coal concentration in the feed slurry was fixed at 27 wt.%. Hydrogen treatrate was ca. 15 wt.% based on dry coal.

A typical two-stage liquefaction run was of 8-10 hours duration. However, an additional 6-8 hours was required for preliminary and post-run operations. In the majority of runs the controlled variables lined-out about 4-5 hours after start-up. During the steady state operating period, vent gas streams were sampled and other product streams were collected for analytical and material balance purposes. A detailed operating procedure is given in Appendix E.

In these experiments the following definitions for the product fractions isolated by solvent extraction were used:

Oil - pentane solubles

Asphaltenes - toluene solubles, pentane insolubles

Preasphaltenes - THF solubles, toluene insolubles.

A direct extraction technique was used to determine these fractions. Individual samples of product slurry were separately extracted with pentane, toluene and tetrahydrofuran (THF). Conversions were calculated on the basis of maf coal fed to the reaction zone during the steady state sampling period and unconverted coal in the dry insoluble residues. Conversion calculations are described in Appendix A.

Vent gas streams were analyzed using a Perkin-Elmer Sigma 3B gas chromatograph interfaced with a Perkin-Elmer Sigma 10 chromatography data station. Water collected in the high pressure knock-out vessel was analyzed for ammonia and sulphide using standard wet chemical methods.

Results of the two-stage thermal/catalytic experiments with Katalco 550 are summarized in Table 8. Run No. 7001 was the first scheduled test. It was carried out with Prince coal at operating conditions similar to those of Run No. 6027. The latter experiment was conducted in Phase VI. The purpose of Run No. 7001 was to check the activity of the catalyst which had been stored in the second-stage reactor for about four months. Coal conversion, raw product yields and hydrogen consumption from Run No. 7001 were found to be in good agreement with results from Run No. 6027. Thus, the activity of the catalyst was not affected by our handling and storage procedure.

The temperature of the thermal dissolver in Run No. 7001 was 440°C. Coal conversion and pentane soluble oil yield were 93.8 and 53.6% respectively. The dissolver was operated at 430°C with Prince Hub seam coal in Run No. 7004. The decrease of 10°C in the dissolver temperature resulted in a lower oil yield, 49.4%. Hydrogen consumption in Run No. 7004 was 5.2% compared with 6.6% in Run No. 7001.

Run Nos. 7002 and 7006 were conducted with Donkin Harbour seam coal. Operating conditions for Run No. 7002 were selected based on previous experience with Harbour seam coal from the Lingan mine. Results of the test were promising in that coal conversion was high and 50.7% oil yield was achieved compared with 47.0% from Lingan. In Run No. 7006 both thermal dissolver and catalytic reactor temperatures were increased by 5°C. The higher temperature caused a slight increase in gas production but had little effect on conversion of coal to oil.

TABLE 8

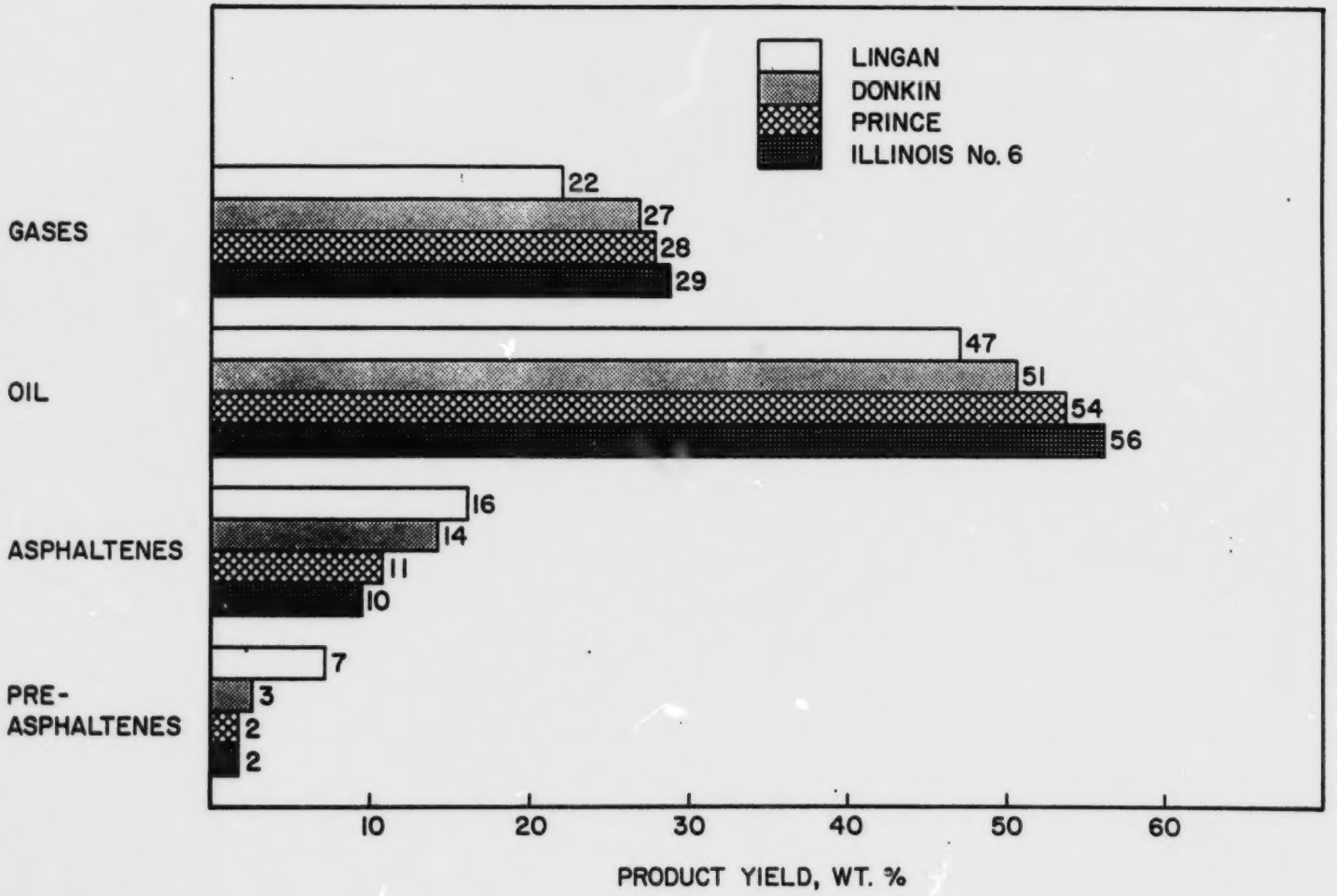
RESULTS FROM TWO-STAGE THERMAL/CATALYTIC LIQUEFACTION TESTS WITH KATALCO 550

RUN NO. COAL TESTED	7001 PRINCE HB-7	7004 PRINCE HB-7	7002 DONKIN HR-7	7006 DONKIN HR-7	7003 ILLINOIS NO. 6	7005 ILLINOIS NO. 6
Temperatures: °C						
Thermal Dissolver	440	430	450	455	440	440
Catalytic Reactor	425	425	425	430	425	425
Back Pressure, MPa	20.68	20.68	20.68	20.68	20.68	20.68
Slurry Feedrate, kg/hr	0.998	1.008	1.000	1.000	1.041	1.002
H_2 Treatrate, Wt. % Dry Coal	15.1	14.9	14.8	15.0	14.3	14.8
Catalyst Age (kg coal/kg catalyst)	117	135	122	145	130	140
CONVERSIONS, WT. % OF maf COAL						
Pentane Solubles plus Gases	81.4	76.6	77.3	78.3	83.3	84.6
Toluene Solubles plus Gases	92.1	90.2	91.4	91.1	93.8	94.1
THF Solubles plus Gases	93.8	93.2	93.9	93.5	95.5	95.8
PRODUCT DISTRIBUTION, WT. % OF maf COAL						
Gases	27.8	27.2	26.6	28.0	29.5	28.5
Oil	53.6	49.4	50.7	50.3	53.8	56.1
Asphaltenes	10.7	13.6	14.1	12.8	10.5	9.5
Preasphaltenes	1.7	3.0	2.5	2.4	1.7	1.7
H_2 CONSUMPTION, WT. % OF maf COAL	6.6	5.2	5.9	6.1	6.1	6.8

Run Nos. 7003 and 7005 were carried out with Illinois No. 6 coal. About 5 hours into Run No. 7003 the high pressure metering pump failed to deliver slurry. The pump was isolated from the system and new packing was installed in the pump body. The metering pump was off-line for about 1.5 hours during which time reactor temperatures were permitted to fall by 80-100°C and the flow of hydrogen was suspended. After reactivating the pump, operations resumed for an additional 6 hours. Target operating conditions were reached about 1 hour after the restart. Run No. 7005 was made to assess the validity of the data obtained from Run No. 7003. Coal conversion was high in both runs, ca. 96%. However, oil yield and hydrogen consumption were slightly higher in Run No. 7005 at 56.1 and 6.8% respectively.

Figure 3 illustrates raw product distribution as a function of coal feedstock using Katalco 550 catalyst. Data for Lingan coal is from Phase VI, Run No. 6026. Product distribution varied considerably as a function of coal feedstock. Illinois No. 6 yielded the lightest product distribution in terms of oil, asphaltenes and preasphaltenes. Among the Sydney area coals, Prince Hub seam produced more oil and less asphaltenes than the Harbour seam samples.

Figure 3 PRODUCT DISTRIBUTION AS A FUNCTION OF COAL FEEDSTOCK WITH KATALCO 550 AT 15 WT. % HYDROGEN TREATRATE



6.2 Operations Using Amocat 1C Bimodal Catalyst

Amocat 1C is an alumina-supported nickel-molybdenum catalyst. It has a bimodal pore size distribution with average pore diameters of 100 and 1500A°.

Prior to initiating coal liquefaction experiments, the fresh catalyst was sulphided in-situ. This was accomplished using hydrogenated anthracene oil, 7.5%H, spiked with dimethyl disulphide. The sulphiding procedure was carried out at 17.24 MPa and a start-of-run temperature of 175°C. The catalyst was sulphided over a 30-hour period increasing reactor temperature gradually to a maximum of 370°C.

The first liquefaction run with Amocat 1C, Run No. 7007, was of 24 hours duration with Lingan coal to condition the catalyst. The run was conducted at a slurry feedrate of 1.5 kg/hr, a total pressure of 17.24 MPa and thermal/catalytic reactor temperatures of 450 and 425°C respectively. Details pertaining to operating conditions and performance are presented in Appendix B. Material balance data is given in Appendix C.

After completion of the catalyst conditioning operation, a total of eleven experiments were carried out in a once-through mode with Lingan Harbour, Prince Hub, Donkin Harbour and Illinois No. 6 coals. The tests were conducted varying thermal dissolver and catalytic reactor temperatures, hydrogen treatrate and coal concentration in the feed slurry. A summary of the results from these experiments are presented in Table 9.

TABLE 9

RESULTS FROM TWO-STAGE THERMAL/CATALYTIC LIQUEFACTION TESTS WITH AMOCAT 1C

RUN NO. COAL TESTED	7010 LINGAN HR-7	7015 LINGAN HR-7	7018 LINGAN HR-7	7011 DONKIN HR-7	7012 DONKIN HR-7	7014 DONKIN HR-7
Temperatures: °C						
Thermal Dissolver	450	450	460	450	450	450
Catalytic Reactor	425	425	435	425	400	425
Back Pressure, MPa	20.68	20.68	20.68	20.68	20.68	20.68
Slurry Feedrate, kg/hr	1.043	1.007	1.018	1.015	1.017	0.982
H_2 Treatrate, Wt. % Dry Coal	14.4	24.8	24.5	14.8	14.8	25.5
Catalyst Age (kg coal/kg catalyst)	52	85	112	59	68	76
CONVERSIONS, WT. % OF maf COAL						
Pentane Solubles plus Gases	74.2	74.5	79.6	79.6	69.3	81.2
Toluene Solubles plus Gases	90.9	91.0	92.2	92.4	89.8	92.6
THF Solubles plus Gases	94.0	94.1	94.5	93.9	93.3	93.9
PRODUCT DISTRIBUTION, WT. % OF maf COAL						
Gases	22.4	25.2	31.1	25.3	20.8	27.7
Oil	51.8	49.3	48.5	54.3	48.5	53.5
Asphaltenes	16.7	16.5	12.6	12.8	20.5	11.4
Preasphaltenes	3.1	3.1	2.3	1.5	3.5	1.3
H_2 CONSUMPTION, WT. % OF maf COAL	5.7	6.6	7.6	6.3	5.1	8.3

TABLE 9 (Continued)

RESULTS FROM TWO-STAGE THERMAL/CATALYTIC LIQUEFACTION TESTS WITH AMOCAT 1C

RUN NO. COAL TESTED	7008 PRINCE HB-7	7009 PRINCE HB-7	7016 PRINCE HB-7	7019 PRINCE HB-7	7017 ILLINOIS NO. 6
Temperatures: °C					
Thermal Dissolver	450	440	440	440	440
Catalytic Reactor	425	425	425	425	425
Back Pressure, MPa	20.68	20.68	20.68	20.68	20.68
Slurry Feedrate, kg/hr	1.002	0.970	1.007	1.030	1.062
H_2 Treatrate, Wt. % Dry Coal	15.1	15.6	24.8	23.9	23.5
Catalyst Age (kg coal/kg catalyst)	37	45	94	122	103
CONVERSIONS, WT. % OF maf COAL					
Pentane Solubles plus Gases	84.8	83.5	82.6	79.8	87.7
Toluene Solubles plus Gases	92.9	92.5	92.2	91.1	93.7
THF Solubles plus Gases	93.4	93.4	93.2	93.0	95.1
PRODUCT DISTRIBUTION, WT. % OF maf COAL					
Gases	28.6	27.2	30.2	27.6	29.2
Oil	56.2	56.3	52.4	52.2	58.5
Asphaltenes	8.1	9.0	9.6	11.3	6.0
Preasphaltenes	0.5	0.9	1.0	1.9	1.4
H_2 CONSUMPTION, WT. % OF maf COAL	8.2	7.6	7.1	6.6	8.7

Run Nos. 7010, 7015 and 7018 were carried out with Lingan Harbour seam coal varying hydrogen treatrate and the temperature of both reactors. High coal conversion, ca. 94%, was achieved in all three runs. In Run Nos. 7010 and 7015 first and second-stage reactor temperatures were fixed at 450 and 425°C respectively. Increasing the hydrogen treatrate from ca. 15 wt.% in Run No. 7010 to ca. 25 wt.% in Run No. 7015 influenced the following shift in product distribution and hydrogen consumption:

- gases increased from 22.4 to 25.2%
- oil decreased from 51.8 to 49.3%
- hydrogen consumption increased from 5.7 to 6.6%.

In Run No. 7018, thermal dissolver temperature and catalytic reactor temperature were raised 10°C. Operating both stages at higher temperature resulted in higher hydrogen consumption and gas production. Oil yield, however, remained essentially the same.

Run Nos. 7011, 7012 and 7014 were made with Donkin Harbour seam coal. Significant changes in product yields were observed when the second-stage reactor temperature was decreased from 425°C in Run No. 7011 to 400°C in Run No. 7012. Results were as follows:

- gases decreased from 25.3 to 20.8%
- oil decreased from 54.3 to 48.5%
- asphaltenes increased from 12.8 to 20.5%
- preasphaltenes increased from 1.5 to 3.5%
- hydrogen consumption decreased from 6.3 to 5.1%.

A comparison of Run No. 7011 with Run No. 7014 illustrates the effect of increasing the hydrogen treatrate from 15 to 25 wt.%. Hydrogen

consumption, gas production and oil yields exhibited directional trends similar to the ones observed with Lingan coal.

Prince Hub seam coal was tested in Run Nos. 7008, 7009, 7016 and 7019. The experiments were conducted varying thermal dissolver temperature, hydrogen treatrate and coal concentration. Catalytic reactor temperature was fixed at 425°C. Coal conversion was high, ca. 93%, in all four runs while oil yields ranged from 52.2 to 56.3%.

An examination of the results from Run Nos. 7008 and 7009 indicates that dissolver temperature over the range of 440 to 450°C has little effect on raw product yields. However, gas production and hydrogen consumption are slightly lower at 440°C.

Hydrogen treatrate was increased to 25 wt.% in Run No. 7016. A comparison of data from Run No. 7009, 15 wt.% hydrogen treatrate, and Run No. 7016 shows the following shift in product distribution:

- gases increased from 27.2 to 30.2%
- oil decreased from 56.3 to 52.4%

The effect of higher coal concentration in the feed slurry was investigated in Run No. 7019. Increasing the coal concentration from 27 wt.% in Run No. 7016 to 30 wt.% in Run No. 7019 had little effect on oil yield. Furthermore, lower levels of hydrogen consumption and gas production were observed in the latter test.

Run No. 7017 was conducted with Illinois No. 6 coal at operating conditions similar to Run No. 7016. This run produced the highest coal conversion, 95.1%, and the highest oil yield, 58.5%. The yield of asphaltenes was correspondingly low, 6.0%. Hydrogen consumption was measured at 8.7%.

Figures 4 and 5 illustrate raw product distribution as a function of coal feedstock using Amocat 1C at 15 and 25 wt.% hydrogen treatrate. In general, directional trends with regard to yields of gases, oil, asphaltenes and preasphaltenes are similar to those observed with Katalco 550. The order of coal reactivity established in this series of tests is as follows: Illinois No. 6 > Prince Hub > Donkin Harbour > Lingan Harbour.

The difference in coal reactivity is primarily attributed to coal rank. Simple correlations show the dependence of coal reactivity on rank, defined in terms of maximum reflectance of vitrinite (%R_o max.) and composition in terms of reflectance types. For example, the most reactive coal, Illinois No. 6, has the lowest vitrinite reflectance, 0.62, and the lowest reflectance types, V-5 and V-6.

Figure 4 PRODUCT DISTRIBUTION AS A FUNCTION OF COAL FEEDSTOCK WITH AMOCAT 1C AT 15 WT. % HYDROGEN TREATRATE

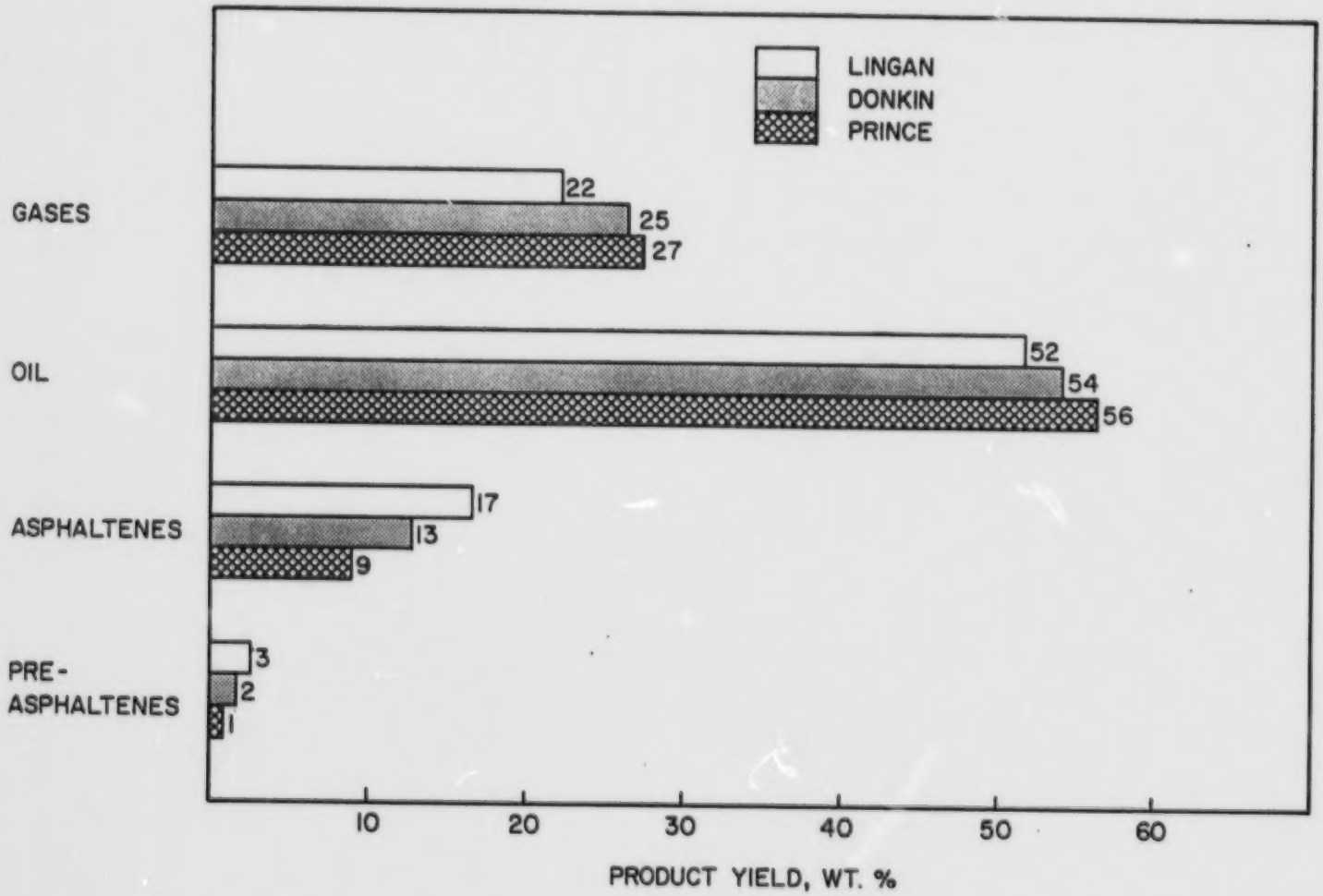
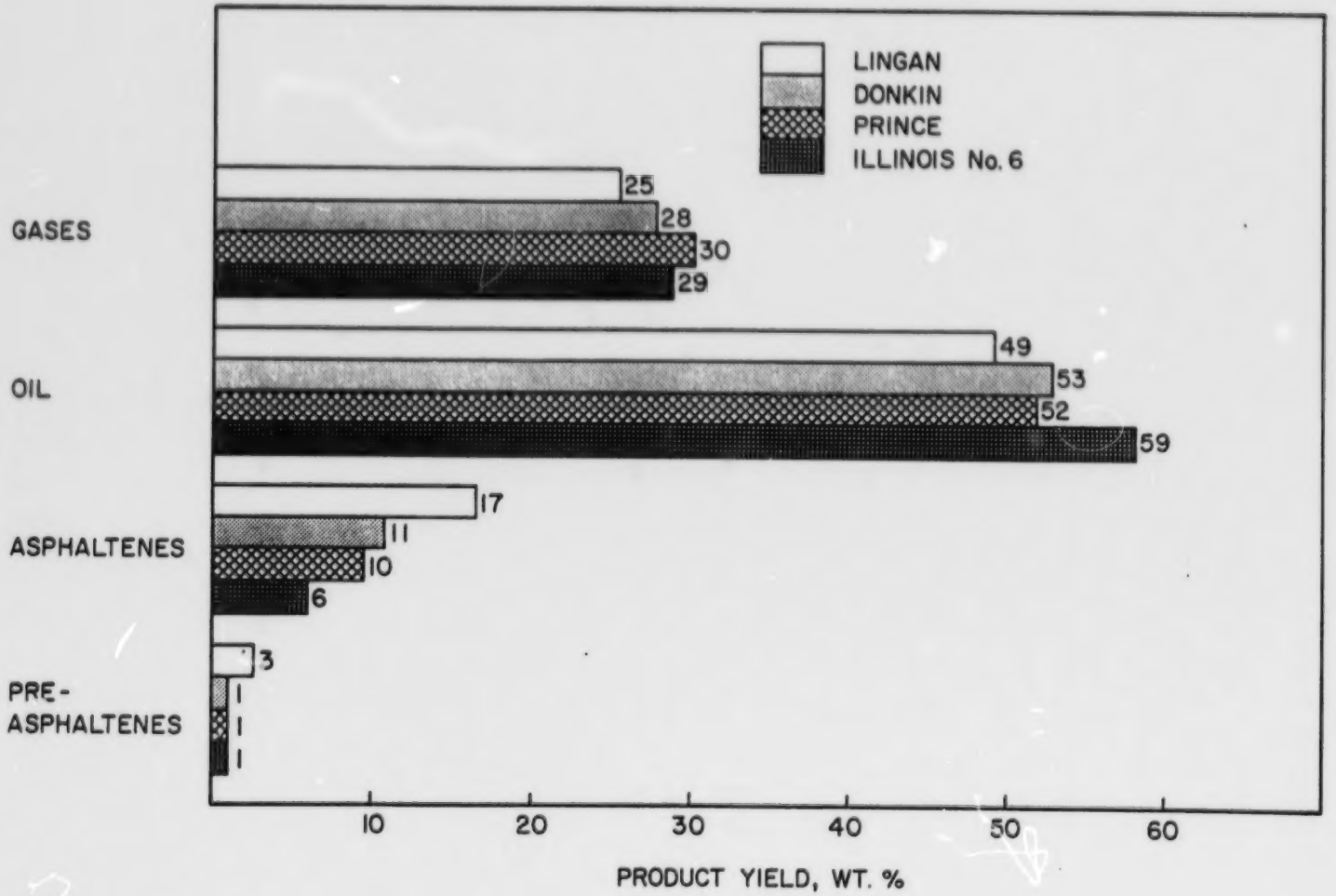


Figure 5 PRODUCT DISTRIBUTION AS A FUNCTION OF COAL FEEDSTOCK WITH AMOCAT 1C AT 25 WT. % HYDROGEN TREATRATE



6.3 Comparison of Unimodal and Bimodal Catalyst Performance

The evaluation of catalysts is an essential part of our program on two-stage thermal/catalytic coal liquefaction. Recently, experimental work has been concentrated on evaluating the performance of a unimodal catalyst, Katalco 550, and a bimodal catalyst, Amocat 1C, with three Sydney area coals. Katalco 550 is a nickel-tungsten catalyst, whereas, Amocat 1C is a nickel-molybdenum catalyst. Nevertheless, the performance comparison made herein is based on pore size distribution of the catalyst not metal type. Earlier studies with the nickel-tungsten catalyst and a unimodal nickel-molybdenum catalyst, Katalco 504-K, showed that both micropore catalysts produced very similar yields of raw products from Lingan coal (7).

Coal conversion and raw product distribution were used as parameters in evaluating the performance of the catalysts. Direct comparisons were made between runs conducted at similar operating conditions; for example, Run Nos. 7001 and 7009 in the case of Prince Hub seam coal. Run No. 7010, with Amocat 1C and Lingan coal, was compared with Run No. 6026 from Phase VI.

High coal conversion, 92 - 94%, was achieved with both catalysts over the range of operating conditions investigated. Product yields are illustrated for Lingan Harbour, Donkin Harbour and Prince Hub seam coals in Figure 6. Experiments with the bimodal catalyst produced higher oil yields and lower levels of asphaltene/preasphaltene

components than tests with the unimodal catalyst. These results suggest that the bimodal pore size distribution is superior for conversion of large coal-derived molecules to lighter components.

Elemental analysis of pentane, toluene and THF soluble fractions from all experiments are presented in Table 10. A comparison of the hydrogen, nitrogen and sulphur levels in the fractions from Run Nos. 7001 and 7009 suggest that Amocat 1C is more active for hydrogenation and denitrogenation reactions. Whereas, Katalco 550 is more active for desulphurization. Such activity trends observed in comparatively fresh catalysts, however, may not be indicative of the relative performances of the catalysts after ageing.

Figure 6 COMPARISON OF PRODUCT YIELDS WITH KATALCO 550 AND AMOCAT IC

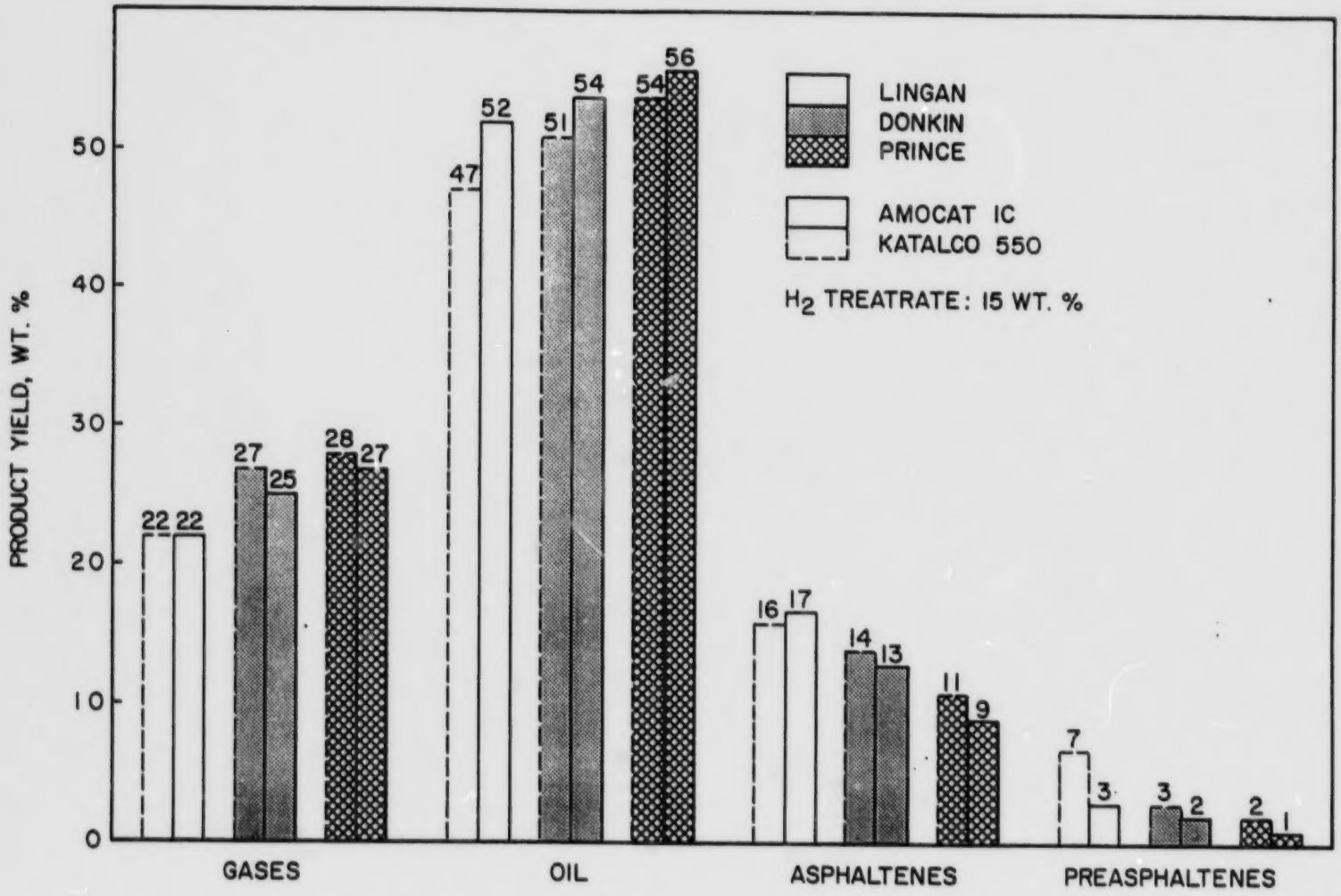


TABLE 10

ELEMENTAL ANALYSIS ON PENTANE, TOLUENE AND THF SOLUBLE FRACTIONS FROM TWO-STAGE LIQUEFACTION TESTS

RUN NO.	7001	7002	7003	7004	7005	7006	7007	7008	7009	7010
Pentane Soluble Fraction										
Carbon, Wt. %	89.73	90.56	89.96	90.04	90.15	89.60	90.07	90.08	90.56	89.92
Hydrogen, Wt. %	9.15	8.90	9.13	9.05	9.17	8.98	9.12	9.14	9.35	9.04
Nitrogen, Wt. %	0.17	0.19	0.19	0.19	0.17	0.18	0.21	0.11	0.15	0.16
Sulphur, Wt. %	0.042	0.038	0.042	0.032	0.042	0.027	0.021	0.054	0.051	0.020
Toluene Soluble Fraction										
Carbon, Wt. %	90.10	90.40	89.77	89.95	89.39	89.70	89.87	89.69	90.65	89.59
Hydrogen, Wt. %	8.94	8.70	8.91	8.92	8.87	8.92	8.97	8.98	9.11	8.71
Nitrogen, Wt. %	0.20	0.23	0.22	0.21	0.22	0.24	0.30	0.15	0.15	0.22
Sulphur, Wt. %	0.043	0.040	0.056	0.048	0.055	0.042	0.023	0.075	0.075	0.026
THF Soluble Fraction										
Carbon, Wt. %	89.84	89.95	89.90	89.89	89.56	89.86	89.99	90.19	90.10	89.92
Hydrogen, Wt. %	8.97	8.77	8.94	8.98	9.00	8.86	8.99	9.05	9.23	8.78
Nitrogen, Wt. %	0.22	0.26	0.23	0.23	0.24	0.26	0.35	0.18	0.15	0.26
Sulphur, Wt. %	0.067	0.069	0.085	0.072	0.102	0.067	0.029	0.096	0.093	0.028

Katalco 550 Nickel-Tungsten Catalyst Used in Run Nos. 7001-7006.

Amocat 1C Nickel-Molybdenum Catalyst Used in Run Nos. 7007-7019.

TABLE 10 (Continued)

ELEMENTAL ANALYSIS ON PENTANE, TOLUENE AND THF SOLUBLE FRACTIONS FROM TWO-STAGE LIQUEFACTION TESTS

RUN NO.	7011	7012	7014	7015	7016	7017	7018	7019
Pentane Soluble Fraction								
Carbon, Wt. %	90.14	90.09	89.02	90.23	89.34	90.36	90.16	90.15
Hydrogen, Wt. %	9.12	8.99	9.02	9.15	8.94	9.21	9.15	9.20
Nitrogen, Wt. %	0.16	0.20	0.16	0.20	0.15	0.14	0.21	0.15
Sulphur, Wt. %	0.037	0.038	0.028	0.010	0.023	0.038	0.014	0.037
Toluene Soluble Fraction								
Carbon, Wt. %	90.11	90.28	89.91	90.16	88.47	90.49	90.19	89.97
Hydrogen, Wt. %	8.99	8.97	8.99	8.96	8.82	9.15	9.03	9.03
Nitrogen, Wt. %	0.22	0.27	0.19	0.26	0.18	0.19	0.25	0.22
Sulphur, Wt. %	0.054	0.057	0.035	0.016	0.030	0.051	0.019	0.049
THF Soluble Fraction								
Carbon, Wt. %	89.73	90.18	90.35	90.35	88.55	90.14	89.96	89.75
Hydrogen, Wt. %	8.83	8.91	9.00	8.95	8.84	9.14	8.93	8.83
Nitrogen, Wt. %	0.22	0.29	0.22	0.27	0.18	0.21	0.25	0.23
Sulphur, Wt. %	0.058	0.065	0.051	0.019	0.047	0.064	0.028	0.061

6.4 Recycle Liquefaction of Prince Hub Seam Coal

The short term liquefaction experiments described in Sections 6.1 and 6.2 are useful to investigate variables such as coal reactivity and catalyst performance. However, it is necessary to run longer tests with liquid recycle to determine process yields and product composition. On the basis of the results from the exploratory tests, a recycle liquefaction run was undertaken with Prince Hub seam coal and Amocat 1C nickel-molybdenum catalyst. The recycle liquefaction run, RCL-7020, was of 6 days duration.

Operating conditions for RCL-7020 are shown in Table 11. Catalyst age at the start-of-run was 122 kg coal/kg catalyst. The concentration of coal in the feed slurry was 28 wt.%.

The operational configuration for this run is presented in Figure 7. Hydrogenated anthracene oil, 9 wt.%H, was used as start-up oil. Separator bottoms product was collected over 12-hour operating periods and then processed batchwise. Vacuum filtration was used to recover liquid from the separator bottoms. The recovered liquid was topped at 240°C by atmospheric distillation. Mid-heavy liquid in excess of the quantity required to mix with the feed coal was taken as net product.

Run plots showing coal conversion, C_1 - C_3 gas production, total gas make and hydrogen consumption for RCL-7020 are presented in Figure 8. A summary of performance and material balance data for each 12-hour operating period is given in Table 12.

TABLE 11

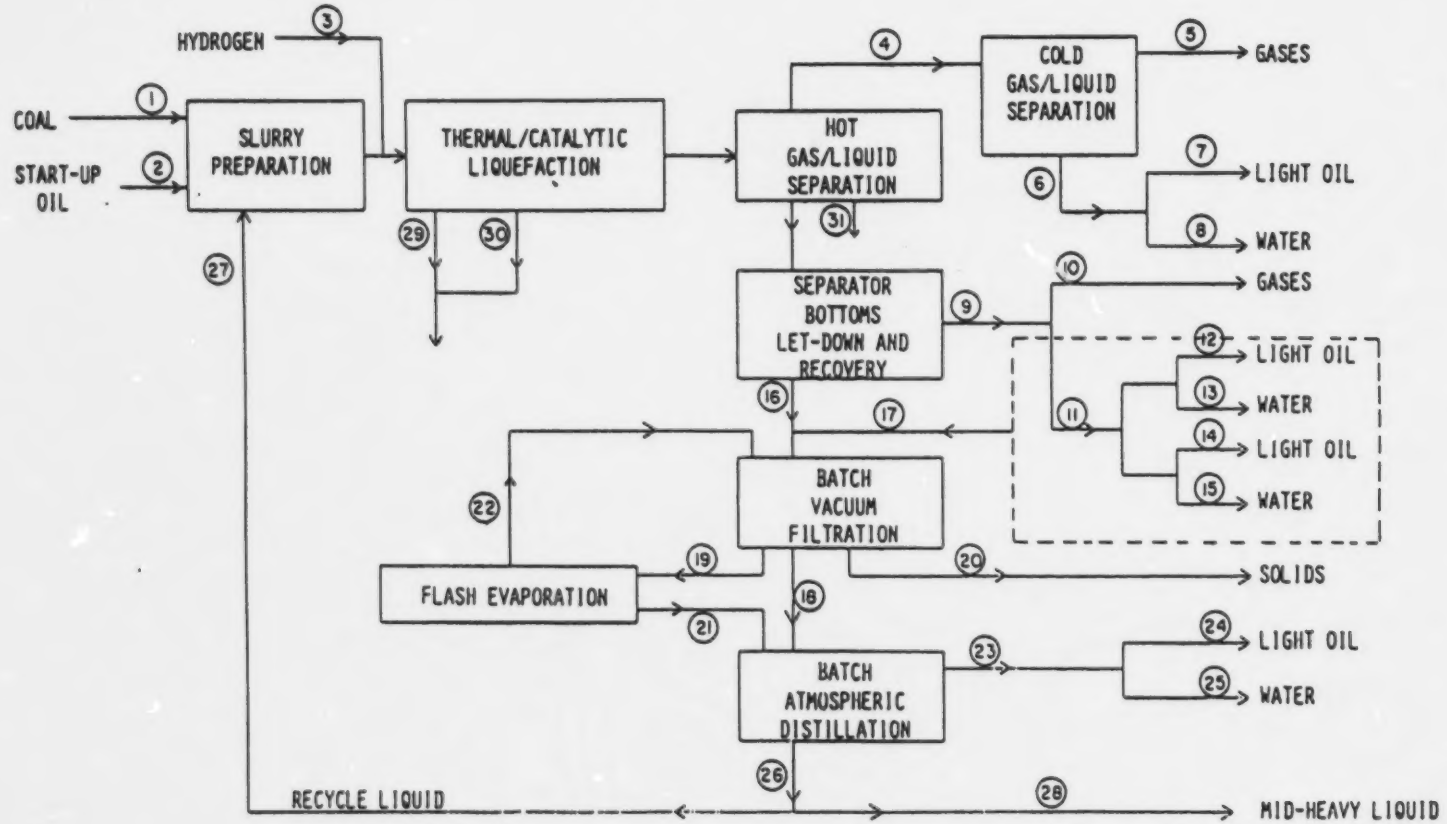
SUMMARY OF OPERATING CONDITIONS FOR RCL-7020

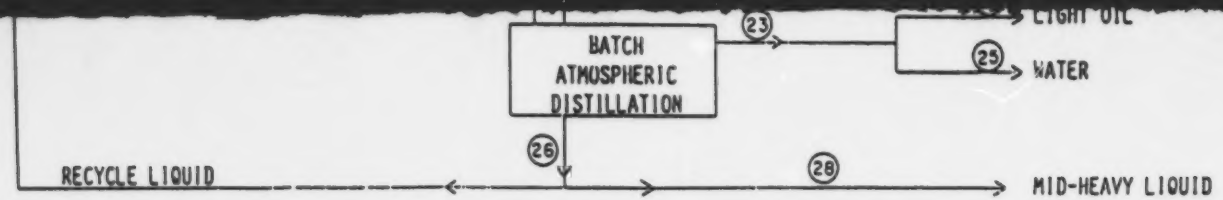
COAL: Prince Hub

CATALYST: Amocat 1C NiMo

OPERATING CONDITIONS	HOURS ON STREAM	
	0-108	108-144
Temperatures, °C:		
Thermal Dissolver	440	440
Catalytic Reactor	425	435
Back Pressure, MPa	20.68	20.68
SHSV, hr^{-1}:		
Thermal Dissolver	1.00	1.00
Catalytic Reactor	0.33	0.33
Coal Concentration, Wt. %	28	28
H_2 Treatrate, Wt. % Dry Coal	20.0	20.0

Figure 7 - OPERATIONAL CONFIGURATION FOR RECYCLE LIQUEFACTION RUN





STREAM NO.	DESCRIPTION
1	Coal
2	Start-Up Oil
3	Hydrogen
4	Hot Separator Overheads
5	Primary Vent Gas
6	High Pressure Knock-Out Bottoms
7	High Pressure Knock-Out - Light Oil
8	High Pressure Knock-Out - Water
9	Low Pressure Product Receiver Overheads
10	Secondary Vent Gas
11	Low Pressure Product Receiver Overhead Condensate
12	Separator Bottoms Flash Knock-Out No. 1 - Light Oil
13	Separator Bottoms Flash Knock-Out No. 1 - Water
14	Separator Bottoms Flash Knock-Out No. 2 - Light Oil
15	Separator Bottoms Flash Knock-Out No. 2 - Water
16	Flashed Separator Bottoms
17	Condensate from SBFKO No. 1 and No. 2
18	Filtered Separator Bottoms
19	Cyclohexane Wash
20	Washed Filter Cake
21	Cyclohexane Solubles Recovered from Filter Cake
22	Recycled Cyclohexane
23	Atmospheric Still Overheads
24	Atmospheric Still Light Oil
25	Atmospheric Still Water
26	Atmospheric Still Bottoms
27	Recycle Liquid
28	Mid-Heavy Liquid Product
29	Thermal Dissolver Contents
30	Catalytic Reactor Contents
31	Separator No. 1 Contents

Figure 8 - RUN PLOTS OF COAL CONVERSION, C_1 - C_3 GAS PRODUCTION, TOTAL GAS MAKE AND HYDROGEN CONSUMPTION FOR RCL-7020

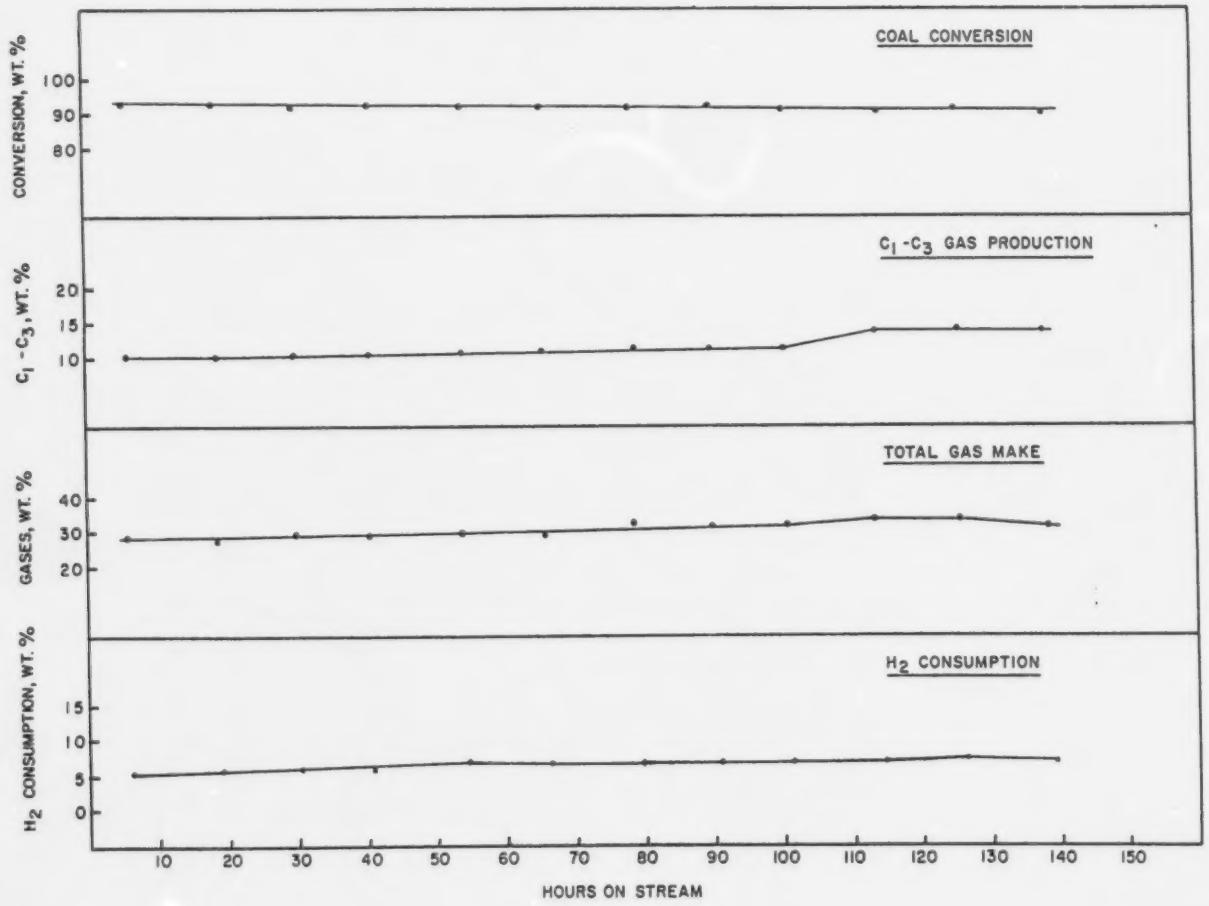


TABLE 12

PERFORMANCE AND MATERIAL BALANCE DATA FOR RCL-7020

OPERATING PERIOD (HRS.)	COAL CONVERSION (WT. % maf COAL)	C_1-C_3 (WT. % maf COAL)	TOTAL GASES (WT. % maf COAL)	HYDROGEN CONSUMPTION (WT. % maf COAL)	MATERIAL BALANCE	
					LIQUEFACTION STEP (WT. %)	OVERALL PROCESS (WT. %)
0-12	92.9	10.2	29.2	5.7	101.7	99.2
12-24	92.7	10.2	28.7	6.1	99.5	97.4
24-36	91.9	11.0	29.6	6.1	99.1	98.6
36-48	92.5	10.6	29.3	6.5	99.4	98.9
48-60	91.7	11.0	29.9	7.2	99.9	99.4
60-72	91.8	11.3	29.1	6.8	99.1	98.9
72-84	91.4	12.2	31.4	6.9	99.3	96.4
84-96	91.7	12.3	31.3	6.9	99.5	NA
96-108	91.4	12.4	31.1	6.9	100.1	100.0
108-120	90.9	14.3	33.7	7.1	102.7	102.7
120-132	91.8	14.4	33.9	7.5	97.5	96.6
132-144	91.3	14.0	32.0	7.2	99.3	100.3

Coal conversion was high over the 6-day period, ranging from 90.9 - 92.7%. A slight downward trend in coal conversion was observed as the run progressed.

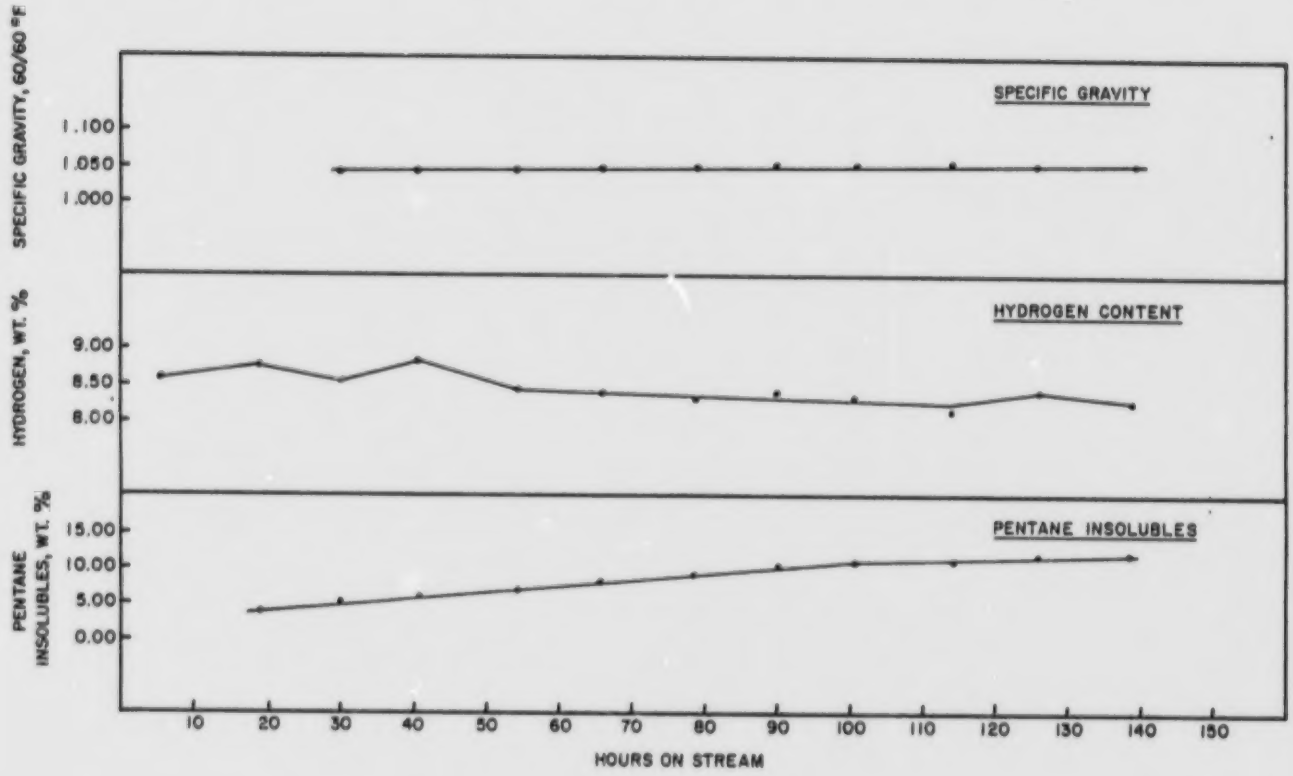
C_1 - C_3 gas production was 10.2% during the first 24 hours but it increased gradually to 12.4% over the next 84 hours. Increasing the catalytic reactor temperature from 425 to 435°C at hour 108 resulted in C_1 - C_3 gas production rising to 14.3%.

Total gas make started at 29.2%. It increased gradually to 31.4% by day 4, then rose to a maximum of 33.9% after the second-stage reactor temperature was increased. In the last 12-hour period total gas production averaged 32.0%.

Hydrogen consumption was measured at 5.7% during the first 12 hours on-stream. It increased to ca. 7% over the next 48 hours and remained at that level for about 3 days. Hydrogen consumption reached a maximum of 7.5% after the catalytic reactor temperature was raised but it slipped back to 7.2% by the end of the run.

Physical and chemical characteristics of the recycle liquid were monitored throughout RCL-7020. Run plots in Figures 9 and 10 show the response of recycle liquid properties as a function of time. Hydrogen aromatic/aliphatic distributions are presented in Table 13.

Figure 9 - RESPONSE OF RECYCLE LIQUID SPECIFIC GRAVITY, HYDROGEN CONTENT AND PENTANE INSOLUBLES IN RCL-7020



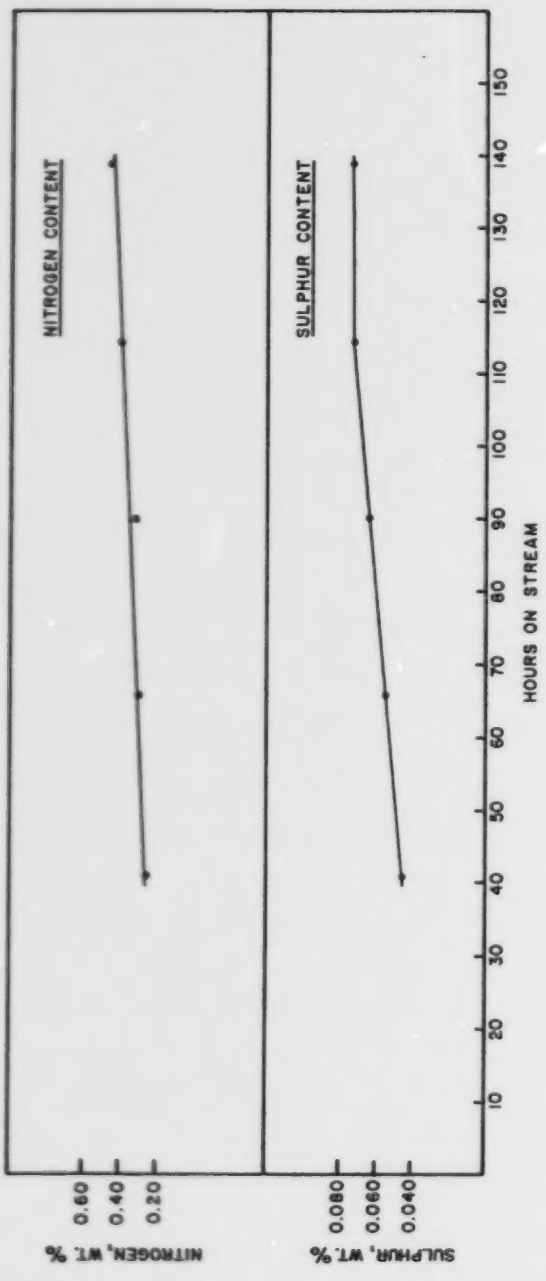


Figure 10 - RESPONSE OF RECYCLE LIQUID NITROGEN CONTENT AND SULPHUR CONTENT IN RCL-7020

TABLE 13

PROPERTIES OF RECYCLE LIQUID FROM RCL-7020

OPERATING PERIOD, HRS.	0-12	12-24	24-36	36-48	48-60	60-72	72-84	84-96	96-108	108-120	120-132	132-144
Specific Gravity, 60/60°F		1.0359	1.0421	1.0441	1.0462	1.0499	1.0530	1.0545	1.0548	1.0565	1.0565	1.0559
Carbon, Wt. %	89.34	89.46	89.13	90.27	89.93	89.75	89.58	89.54	89.51	90.48	89.71	89.53
Hydrogen, Wt. %	8.60	8.77	8.56	8.82	8.47	8.41	8.32	8.42	8.35	8.20	8.45	8.31
Nitrogen, Wt. %				0.28		0.33		0.35		0.41		0.45
Sulphur, Wt. %				0.048		0.057		0.065		0.074		0.076
Pentane Insolubles, Wt. %		3.90	5.15	6.06	7.46	8.01	9.38	10.12	10.84	10.94	11.92	12.10
Proton Distribution, %												
H_{ar} (9-6 ppm)		27.42		27.11		28.07		29.44		29.23		30.02
H_{α} (3.5-2 ppm)		26.08		25.73		26.03		25.86		26.19		26.14
H_{β} (2-1.1 ppm)		35.05		33.99		32.76		29.92		30.79		29.60
H_{γ} (1.1-0.4 ppm)		11.45		13.18		13.15		14.78		13.80		14.25
Distillation Data, Wt. %: (Spinning Band)												
IBP - 205°C												1.6
205 - 335												41.7
335 - 420												25.5
420 - 525°C												7.1
RESID												23.9

Examination of the data indicates that the composition of the recycle liquid has not equilibrated. Increases in specific gravity, pentane insolubles and sulphur content appear to be levelling off, whereas, the nitrogen content is continuing to rise. The hydrogen content appears to be decreasing slightly as the aromatics content of the recycle solvent increases. An apparent decline in the H_β level is expected to reduce solvent quality.

Performance data and net liquid product properties for the last 12-hour operating period are shown in Table 14. Coal conversion over the 132 - 144 hour period averaged 91.3%. The net C_4^+ liquid yield was 67.0%. Inspections were carried out on the C_5^+ liquid product. This liquid has a specific gravity of 0.8976 and is rich in light and middle distillates. The yields of naphtha and light gas oil were 43.5 and 38.1 wt.% respectively.

TABLE 14

PERFORMANCE DATA AND NET LIQUID PRODUCT PROPERTIES FOR RCL-7020

Coal Conversion, Wt. %	91.3
Gas Make, Wt. %	32.0
C_4^+ Liquid Yield, Wt. %	67.0
C_5^+ Liquid Yield, Wt. %	62.8
H_2 Consumption, Wt. %	7.2
C_5^+ Liquid Product Inspections:	
Specific Gravity, 60/60°F	0.8976
Carbon, Wt. %	88.21
Hydrogen, Wt. %	10.69
Nitrogen, Wt. %	0.25
Oxygen, Wt. %	0.78
Sulphur, Wt. %	0.07
Pentane Insolubles, Wt. %	3.01
Distillation Data, °C: (ASTM D-1160)	
IBP	47
10% V/20	89/102
30/40	122/174
50/60	206/226
70/80	236/283
90/95	372/-
EP	523 (94% V)
Distillation Data, Wt. %: (Spinning Band)	
IBP - 205°C	43.5
205 - 335	38.1
335 - 420	8.0
420 - 525°C	1.7
RESID	8.7

CONCLUSIONS AND RECOMMENDATIONS

Phase VII of a research program on the direct liquefaction of Nova Scotia bituminous coals has been completed. This phase of work was concentrated on integrated two-stage thermal/catalytic liquefaction with direct coupling of the first and second-stage reactors. Bench-scale studies were conducted to evaluate the performance of unimodal and bimodal hydroprocessing catalysts with Sydney area coals. Experiments were conducted in once-through and recycle liquefaction process modes.

Test work initiated in Phase VI with Katalco 550 nickel-tungsten catalyst has been completed. Six experiments were carried out in a once-through mode using hydrogenated anthracene oil, 9%H, as reaction solvent. High coal conversions, 93 - 96%, were achieved with Donkin Harbour, Prince Hub and Illinois No. 6 coals. Pentane soluble oil yields for the three coals were 51, 54 and 56% respectively.

A total of twelve experiments were conducted with Amocat 1C nickel-molybdenum catalyst in a once-through mode. The runs were made varying coal feedstock, first-stage thermal dissolver temperature, second-stage catalytic reactor temperature, hydrogen treatrate and coal concentration in the feed slurry. Four coals were tested: Lingan Harbour, Donkin Harbour, Prince Hub and Illinois No. 6.

Operations with Lingan Harbour seam coal at 25 wt.% hydrogen treatrate produced 94% coal conversion and 49% oil yield with the

thermal dissolver at 450°C and the catalytic reactor at 425°C. Increasing the temperature of both reactors 10°C caused gas production and hydrogen consumption to increase while oil yield remained essentially the same. Gas production increased from 25 to 31% whereas hydrogen consumption rose from 6.6 to 7.6%.

In the case of Donkin Harbour seam coal, conversion and oil yield were 94 and 54% respectively at 15 wt.% hydrogen treatrate with the first-stage reactor at 450°C and the second-stage reactor at 425°C. Decreasing the temperature of the catalytic reactor from 425 to 400°C resulted in lower gas and oil production and higher yields of asphaltenes and preasphaltenes.

The preferred operating temperature for the thermal dissolver with Prince Hub seam coal was 440°C. A run conducted at 15 wt.% hydrogen treatrate with the catalytic reactor at 425°C produced 93% coal conversion and 56% oil yield. Gas production decreased from 30 to 27% and hydrogen consumption declined from 7.1 to 6.6% when coal concentration in the feed slurry was increased from 27 to 30 wt.%.

Hydrogen treatrate over the range of 15 to 25 wt.% had little effect on product yields. In general, hydrogen consumption and gas production were higher and oil yields were lower at 25 wt.% hydrogen treatrate.

The order of coal reactivity established is as follows: Illinois No. 6 > Prince Hub > Donkin Harbour > Lingan Harbour. Illinois No. 6

coal yielded the lightest product distribution in terms of oil, asphaltenes and preasphaltenes. The difference in reactivity is primarily attributed to coal rank. Simple correlations show the dependence of coal reactivity on rank, defined in terms of maximum reflectance of vitrinite (%R_o max.) and composition in terms of reflectance types. For example, the most reactive coal, Illinois No. 6, has the lowest vitrinite reflectance, 0.62, and the lowest reflectance types, V-5 and V-6.

Experiments with Katalco 550 and Amocat 1C produced high coal conversions, 92 - 95%, over the range of operating conditions investigated. However, Amocat 1C produced higher oil yields and lower levels of asphaltene-preasphaltene components than Katalco 550. A comparison of test results with Katalco 504-K, Katalco 550 and Amocat 1C shows that the bimodal pore diameter distribution of Amocat 1C is superior for conversion of coal to distillate products.

A 6-day recycle liquefaction run was successfully completed with Prince Hub seam coal using the Amocat 1C bimodal catalyst. Coal conversion averaged 91% over the last 12-hour operating period. The net C_4^+ liquid yield was 67%. High yields of light and middle distillates were achieved in this test. The yields of naphtha and light gas oil were 44 and 38 wt.% respectively.

Based on the results from this work the following recommendations for future work are proposed:

(a) Extend the bench program on recycle liquefaction of Prince Hub seam coal to further investigate process yields, product composition and recycle solvent behaviour.

(b) On completion of the recycle liquefaction test work, convert the thermal dissolver to a catalytic dissolver.

(c) Following this, investigate the potential of catalytic two-stage processing applied to Sydney area coals.

(d) Finally, with the most promising combination of coal and process configuration, conduct long term recycle liquefaction runs to determine ultimate yields, product composition and catalyst ageing characteristics.

At present, conventional coal liquefaction is not economically viable in Eastern Canada. Technological advances in the areas of process and/or plant hardware are required to improve the economics of converting coal into premium quality liquid fuels. Two-stage direct liquefaction offers the potential to enhance distillate yields and improve hydrogen utilization. However, extensive test work is required on two-stage process configurations applied to Sydney area coals to determine the extent to which these improvements can be realized.

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APPENDIX A

CONVERSION CALCULATIONS

APPENDIX A

CONVERSION CALCULATIONS FOR CONTINUOUS COAL LIQUEFACTION EXPERIMENTS

Calculations are based on operations during the steady state sampling period.

CONVERSIONS, WT.%:

$$\text{Pentane sol. + gases} = \frac{\text{(maf coal charged) - (organic pentane insol.)}}{\text{maf coal charged}} \times 100$$

$$\text{Toluene sol. + gases} = \frac{\text{(maf coal charged) - (organic toluene insol.)}}{\text{maf coal charged}} \times 100$$

$$\text{THF sol. + gases} = \frac{\text{(maf coal charged) - (organic THF insol.)}}{\text{maf coal charged}} \times 100$$

$$\text{Gases} = \frac{\text{product gases}}{\text{maf coal charged}} \times 100$$

DEFINITION OF PRODUCTS

Oil = pentane solubles

Asphaltenes = toluene solubles - pentane solubles

Preasphaltenes = THF solubles - toluene solubles

APPENDIX B

SUMMARY OF OPERATING CONDITIONS AND PERFORMANCE FOR TWO-STAGE THERMAL/CATALYTIC LIQUEFACTION EXPERIMENTS

APPENDIX B

SUMMARY OF OPERATING CONDITIONS AND PERFORMANCE FOR TWO-STAGE THERMAL/CATALYTIC LIQUEFACTION TESTS

RUN NO. COAL TESTED CATALYST TESTED	7001 PRINCE HB-7 KATALCO 550	7002 DONKIN HR-7 KATALCO 550	7003 ILLINOIS NO. 6 KATALCO 550	7004 PRINCE HB-7 KATALCO 550
A. OPERATING CONDITIONS				
Unit back pressure, MPa	20.68	20.68	20.68	20.68
Temperatures, °C:				
Thermal Dissolver	440	450	440	430
Catalytic Reactor	425	425	425	425
Slurry Preheater	240	240	240	240
Hot Separator	300	300	300	300
Feedrates:				
Slurry, kg/hr	0.998	1.000	1.041	1.008
Raw Coal, kg/hr	0.269	0.270	0.281	0.272
Hydrogen, L/hr	436.	438.	432.	432.
SHSV, hr^{-1}; Thermal Dissolver	1.00	1.00	1.00	1.00
Catalytic Reactor	0.33	0.33	0.33	0.33
Hydrogen Treatrate, Wt. % Dry Coal	15.1	14.8	14.3	14.9
Catalyst Age (kg coal/kg catalyst)	117	122	130	135
B. CONVERSIONS OF maf COAL, WT. %				
Pentane Solubles plus Gases	81.4	77.3	83.3	76.6
Toluene Solubles plus Gases	92.1	91.4	93.8	90.2
THF Solubles plus Gases	93.8	93.9	95.5	93.2
C. PRODUCT YIELDS ON maf COAL, WT. %				
CH_4	3.7	4.3	3.6	3.4
C_2H_6	3.9	5.8	3.5	3.8
C_3H_8	2.9	3.7	3.1	2.3
C_4H_{10}	3.1	3.1	3.1	3.0
CO	0.4	0.5	0.7	0.5
H_2S	2.0	1.9	2.9	2.6
H_2O	10.9	6.6	11.8	10.8
NH_3	0.9	0.7	0.8	0.8
Oil	53.6	50.7	53.8	49.4
Asphaltenes	10.7	14.1	10.5	13.6
Preasphaltenes	1.7	2.5	1.7	3.0
Unreacted Coal	6.2	6.1	4.5	6.8
D. H_2 CONSUMPTION, WT. % OF maf COAL	6.6	5.9	6.1	5.2

APPENDIX B

SUMMARY OF OPERATING CONDITIONS AND PERFORMANCE FOR TWO-STAGE THERMAL/CATALYTIC LIQUEFACTION TESTS

RUN NO. COAL TESTED CATALYST TESTED	7005 ILLINOIS NO. 6 KATALCO 550	7006 DONKIN HR-7 KATALCO 550	7007 LINGAN HR-6 AMOCAT 1 C	7008 PRINCE HB-7 AMOCAT 1 C
A. OPERATING CONDITIONS				
Unit back pressure, MPa	20.68	20.68	17.24	20.68
Temperatures, °C:				
Thermal Dissolver	440	455	450	450
Catalytic Reactor	425	430	425	425
Slurry Preheater	240	240	240	240
Hot Separator	300	300	300	300
Feedrates:				
Slurry, kg/hr	1.002	1.000	1.504	1.002
Raw Coal, kg/hr	0.271	0.270	0.406	0.271
Hydrogen, L/hr	432.	444.	444.	438.
SHSV, hr^{-1}; Thermal Dissolver	1.00	1.00	1.50	1.00
Catalytic Reactor	0.33	0.33	0.50	0.33
Hydrogen Treatrate, Wt. % Dry Coal	14.8	15.0	10.0	15.1
Catalyst Age (kg coal/kg catalyst)	140	145	30	37
B. CONVERSIONS OF maf COAL, WT. %				
Pentane Solubles plus Gases	84.6	78.3	61.6	84.8
Toluene Solubles plus Gases	94.1	91.1	84.7	92.9
THF Solubles plus Gases	95.8	93.5	91.5	93.4
C. PRODUCT YIELDS ON maf COAL, WT. %				
CH_4	3.4	5.2	3.7	4.4
C_2H_6	3.6	5.2	3.7	5.1
C_3H_8	3.2	4.2	2.4	3.7
C_4H_{10}	3.0	3.7	2.3	3.1
CO	0.6	0.4	0.2	0.2
H_2S	2.5	1.9	0.6	2.2
H_2O	11.4	6.7	5.7	9.0*
NH_3	0.8	0.7	0.6	0.9
Oil	56.1	50.3	42.4	56.2
Asphaltenes	9.5	12.8	23.1	8.1
Preasphaltenes	1.7	2.4	6.8	0.5
Unreacted Coal	4.2	6.5	8.5	6.6
D. H_2 CONSUMPTION, WT. % OF maf COAL	6.8	6.1	4.6	8.2

* Based on water production for entire run.

APPENDIX B

SUMMARY OF OPERATING CONDITIONS AND PERFORMANCE FOR TWO-STAGE THERMAL/CATALYTIC LIQUEFACTION TESTS

RUN NO. COAL TESTED CATALYST TESTED	7009 PRINCE HB-7 AMOCAT 1 C	7010 LINGAN HR-7 AMOCAT 1 C	7011 DONKIN HR-7 AMOCAT 1 C	7012 DONKIN HR-7 AMOCAT 1 C
A. OPERATING CONDITIONS				
Unit back pressure, MPa	20.68	20.68	20.68	20.68
Temperatures, °C:				
Thermal Dissolver	440	450	450	450
Catalytic Reactor	425	425	425	400
Slurry Preheater	240	240	240	240
Hot Separator	300	300	300	300
Feedrates:				
Slurry, kg/hr	0.970	1.043	1.015	1.017
Raw Coal, kg/hr	0.262	0.282	0.274	0.275
Hydrogen, L/hr	438.	444.	444.	444.
SHSV, hr^{-1}; Thermal Dissolver	1.00	1.00	1.00	1.00
Catalytic Reactor	0.33	0.33	0.33	0.33
Hydrogen Treatrate, Wt. % Dry Coal	15.6	14.4	14.8	14.8
Catalyst Age (kg coal/kg catalyst)	45	52	59	68
B. CONVERSIONS OF maf COAL, WT. %				
Pentane Solubles plus Gases	83.5	74.2	79.6	69.3
Toluene Solubles plus Gases	92.5	90.9	92.4	89.8
THF Solubles plus Gases	93.4	94.0	93.9	93.3
C. PRODUCT YIELDS ON maf COAL, WT. %				
CH_4	3.9	4.3	4.3	3.3
C_2H_6	4.0	4.2	4.2	3.8
C_3H_8	3.2	3.3	3.4	2.6
C_4H_{10}	3.2	3.3	3.1	2.7
CO	0.3	0.2	0.3	0.3
H_2S	2.3	0.6	1.8	1.7
H_2O	9.4*	5.7	7.4	5.8
NH_3	0.9	0.8	0.8	0.6
Oil	56.3	51.8	54.3	48.5
Asphaltenes	9.0	16.7	12.8	20.5
Preasphaltenes	0.9	3.1	1.5	3.5
Unreacted Coal	6.6	6.0	6.1	6.7
D. H_2 CONSUMPTION, WT. % OF maf COAL	7.6	5.7	6.3	5.1

* Based on water production for entire run.

APPENDIX B

SUMMARY OF OPERATING CONDITIONS AND PERFORMANCE FOR TWO-STAGE THERMAL/CATALYTIC LIQUEFACTION TESTS

RUN NO.	7014	7015	7016	7017
COAL TESTED	DONKIN HR-7	LINGAN HR-7	PRINCE HB-7	ILLINOIS NO. 6
CATALYST TESTED	AMOCAT 1 C	AMOCAT 1 C	AMOCAT 1 C	AMOCAT 1 C
A. OPERATING CONDITIONS				
Unit back pressure, MPa	20.68	20.68	20.68	20.68
Temperatures, °C:				
Thermal Dissolver	450	450	440	440
Catalytic Reactor	425	425	425	425
Slurry Preheater	240	240	240	240
Hot Separator	300	300	300	300
Feedrates:				
Slurry, kg/hr	0.982	1.007	1.007	1.062
Raw Coal, kg/hr	0.265	0.272	0.272	0.287
Hydrogen, L/hr	738.	738.	726.	726.
SHSV, hr^{-1}; Thermal Dissolver	1.00	1.00	1.00	1.00
Catalytic Reactor	0.33	0.33	0.33	0.33
Hydrogen Treatrate, Wt. % Dry Coal	25.5	24.8	24.8	23.5
Catalyst Age (kg coal/kg catalyst)	76	85	94	103
B. CONVERSIONS OF maf COAL, WT. %				
Pentane Solubles plus Gases	81.2	74.5	82.6	87.7
Toluene Solubles plus Gases	92.6	91.0	92.2	93.7
THF Solubles plus Gases	93.9	94.1	93.2	95.1
C. PRODUCT YIELDS ON maf COAL, WT. %				
CH_4	4.4	4.8	4.2	3.4
C_2H_6	5.0	4.9	3.8	4.0
C_3H_8	3.1	3.5	3.3	2.9
C_4H_{10}	4.9	4.2	3.9	3.3
CO	0.3	0.9	0.9	0.7
H_2S	2.1	0.7	2.6	3.0
H_2O	6.9	5.2	10.5	10.9
NH_3	1.0	1.0	1.0	1.0
Oil	53.5	49.3	52.4	58.5
Asphaltenes	11.4	16.5	9.6	6.0
Preasphaltenes	1.3	3.1	1.0	1.4
Unreacted Coal	6.1	5.9	6.8	4.9
D. H_2 CONSUMPTION, WT. % OF maf COAL	8.3	6.6	7.1	8.7

APPENDIX B

SUMMARY OF OPERATING CONDITIONS AND PERFORMANCE
FOR TWO-STAGE THERMAL/CATALYTIC LIQUEFACTION TESTS

RUN NO.	7018	7019
COAL TESTED	LINGAN HR-7	PRINCE HB-7
CATALYST TESTED	AMOCAT 1 C	AMOCAT 1 C
A. OPERATING CONDITIONS		
Unit back pressure, MPa	20.68	20.68
Temperatures, °C:		
Thermal Dissolver	460	440
Catalytic Reactor	435	425
Slurry Preheater	240	240
Hot Separator	300	300
Feedrates:		
Slurry, kg/hr	1.018	1.030
Raw Coal, kg/hr	0.275	0.309
Hydrogen, L/hr	738.	792.
SHSV, hr^{-1}; Thermal Dissolver	1.00	1.00
Catalytic Reactor	0.33	0.33
Hydrogen Treatrate, Wt. % Dry Coal	24.5	23.9
Catalyst Age (kg coal/kg catalyst)	112	122
B. CONVERSIONS OF maf COAL, WT. %		
Pentane Solubles plus Gases	79.6	79.8
Toluene Solubles plus Gases	92.2	91.1
THF Solubles plus Gases	94.5	93.0
C. PRODUCT YIELDS ON maf COAL, WT. %		
CH_4	6.3	3.4
C_2H_6	6.9	4.0
C_3H_8	5.1	2.9
C_4H_{10}	4.6	2.9
CO	0.4	0.5
H_2S	0.8	2.7
H_2O	6.0	10.3
NH_3	1.0	0.9
Oil	48.5	52.2
Asphaltenes	12.6	11.3
Preasphaltenes	2.3	1.9
Unreacted Coal	5.5	7.0
D. H_2 CONSUMPTION, WT. % OF maf COAL	7.6	6.6

APPENDIX B

SUMMARY OF OPERATING CONDITIONS AND PERFORMANCE FOR RCL-7020

OPERATING PERIOD, HRS.	0-12	12-24	24-36	36-48
COAL TESTED	PRINCE HB-7	PRINCE HB-7	PRINCE HB-7	PRINCE HB-7
CATALYST TESTED	AMOCAT 1C	AMOCAT 1C	AMOCAT 1C	AMOCAT 1C
A. OPERATING CONDITIONS				
Unit back pressure, MPa	20.68	20.68	20.68	20.68
Temperatures, °C:				
Thermal Dissolver	440	440	440	440
Catalytic Reactor	425	425	425	425
Slurry Preheater	240	240	240	240
Hot Separator	300	300	300	300
Feedrates:				
Slurry, kg/hr	1.074	1.035	1.051	1.047
Raw Coal, kg/hr	0.301	0.290	0.294	0.293
Hydrogen, L/hr	615.	615.	615.	615.
SHSV, hr^{-1}; Thermal Dissolver	1.00	1.00	1.00	1.00
Catalytic Reactor	0.33	0.33	0.33	0.33
Hydrogen Treatrate, Wt. % Dry Coal	19.0	19.7	19.4	19.5
Catalyst Age (kg coal/kg catalyst)	135	146	157	168
B. CONVERSIONS OF maf COAL, WT. %				
THF Solubles plus Gases	92.9	92.7	91.9	92.5
C. PRODUCT YIELDS ON maf COAL, WT. %				
CH_4	3.2	3.4	3.8	3.7
C_2H_6	4.3	3.8	4.1	3.7
C_3H_8	2.7	3.0	3.1	3.2
C_4H_{10}	3.3	3.1	3.3	3.4
CO	0.7	0.7	0.8	0.5
H_2S	2.6	2.6	2.6	2.6
H_2O	11.5	11.2	11.0	11.3
NH_3	0.9	0.9	0.9	0.9
Oil + Asphaltenes + Preasphaltenes	63.7	64.0	62.3	63.2
Unreacted Coal	7.1	7.3	8.1	7.5
D. H_2 CONSUMPTION, WT. % OF maf COAL	5.7	6.1	6.1	6.5

APPENDIX B

SUMMARY OF OPERATING CONDITIONS AND PERFORMANCE FOR RCL-7020

OPERATING PERIOD, HRS.	48-60	60-72	72-84	84-96
COAL TESTED	PRINCE HB-7	PRINCE HB-7	PRINCE HB-7	PRINCE HB-7
CATALYST TESTED	AMOCAT 1C	AMOCAT 1C	AMOCAT 1C	AMOCAT 1C
A. OPERATING CONDITIONS				
Unit back pressure, MPa	20.68	20.68	20.68	20.68
Temperatures, °C:				
Thermal Dissolver	440	440	440	440
Catalytic Reactor	425	425	425	425
Slurry Preheater	240	240	240	240
Hot Separator	300	300	300	300
Feedrates:				
Slurry, kg/hr	1.033	1.033	1.015	1.024
Raw Coal, kg/hr	0.289	0.289	0.284	0.287
Hydrogen, L/hr	615.	615.	615.	615.
SHSV, hr^{-1}; Thermal Dissolver	1.00	1.00	1.00	1.00
Catalytic Reactor	0.33	0.33	0.33	0.33
Hydrogen Treatrate, Wt. % Dry Coal	19.7	19.8	20.1	19.9
Catalyst Age (kg coal/kg catalyst)	179	190	200	210
B. CONVERSIONS OF maf COAL, WT. %				
THF Solubles plus Gases	91.7	91.8	91.4	91.7
C. PRODUCT YIELDS ON maf COAL, WT. %				
CH_4	3.9	4.0	4.1	4.2
C_2H_6	3.8	4.1	4.7	4.7
C_3H_8	3.3	3.2	3.4	3.4
C_4H_{10}	4.1	3.5	3.8	3.8
CO	0.8	0.5	0.6	0.6
H_2S	2.2	2.2	2.6	2.5
H_2O	11.0	10.8	11.3	11.2
NH_3	0.8	0.8	0.9	0.9
Oil + Asphaltenes + Preasphaltenes	61.8	62.7	60.0	60.4
Unreacted Coal	8.3	8.2	8.6	8.3
D. H_2 CONSUMPTION, WT. % OF maf COAL	7.2	6.8	6.9	6.9

APPENDIX B

SUMMARY OF OPERATING CONDITIONS AND PERFORMANCE FOR RCL-7020

OPERATING PERIOD, HRS. COAL TESTED CATALYST TESTED	96-108 PRINCE HB-7 AMOCAT 1C	108-120 PRINCE HB-7 AMOCAT 1C	120-132 PRINCE HB-7 AMOCAT 1C	132-144 PRINCE HB-7 AMOCAT 1C
A. OPERATING CONDITIONS				
Unit back pressure, MPa	20.68	20.68	20.68	20.68
Temperatures, °C:				
Thermal Dissolver	440	440	440	440
Catalytic Reactor	425	435	435	435
Slurry Preheater	240	240	240	240
Hot Separator	300	300	300	300
Feedrates:				
Slurry, kg/hr	1.003	1.018	1.016	1.036
Raw Coal, kg/hr	0.281	0.285	0.284	0.290
Hydrogen, L/hr	615.	615.	615.	615.
SHSV, hr^{-1}; Thermal Dissolver	1.00	1.00	1.00	1.00
Catalytic Reactor	0.33	0.33	0.33	0.33
Hydrogen Treatrate, Wt. % Dry Coal	20.3	20.0	20.1	19.7
Catalyst Age (kg coal/kg catalyst)	220	230	240	251
B. CONVERSIONS OF maf COAL, WT. %				
THF Solubles plus Gases	91.4	90.9	91.8	91.3
C. PRODUCT YIELDS ON maf COAL, WT. %				
CH_4	4.4	5.1	5.3	5.1
C_2H_6	4.4	5.0	5.0	4.9
C_3H_8	3.6	4.2	4.1	4.0
C_4H_{10}	3.8	4.3	4.2	4.2
CO	0.5	0.5	0.8	0.5
H_2S	2.5	2.5	2.6	2.4
H_2O	11.1	11.2	11.0	10.1
NH_3	0.8	0.9	0.9	0.8
Oil + Asphaltenes + Preasphaltenes	60.3	57.2	57.9	59.3
Unreacted Coal	8.6	9.1	8.2	8.7
D. H_2 CONSUMPTION, WT. % OF maf COAL	6.9	7.1	7.5	7.2

APPENDIX C

MATERIAL BALANCES FOR TWO-STAGE THERMAL/CATALYTIC LIQUEFACTION EXPERIMENTS

APPENDIX C

MATERIAL BALANCES FOR TWO-STAGE THERMAL/CATALYTIC LIQUEFACTION TESTS

RUN NO.	7001	7002	7003	7004
COAL TESTED	PRINCE HB-7	DONKIN HR-7	ILLINOIS NO. 6	PRINCE HB-
CATALYST TESTED	KATALCO 550	KATALCO 550	KATALCO 550	KATALCO 55
MOISTURE, WT. %	3.30	1.65	3.03	3.30
ASH, WT. % DRY BASIS	9.10	5.02	11.16	9.10
HOURS ON STREAM	8.62	8.10	11.15	8.00
DURATION OF BALANCE PERIOD, HRS.	2.70	2.02	1.52	1.97
INPUT (g):				
H_2O	24.0	9.0	13.0	17.6
Ash	64.0	26.9	46.3	46.9
maf Coal	640.	509.	368.	469.
Solvent	1967.	1475.	1156.	1442.
H_2	106.4	79.6	59.1	76.6
Total	2801.	2100.	1642.	2052.
OUTPUT (g):				
H_2O	93.6	42.4	56.4	68.2
Ash	46.4	18.3	39.0	35.5
Organic THF Insolubles[1]	39.7	31.2	16.6	32.0
THF Solubles[2]	2396.	1835.	1418.	1769.
H_2	63.9	49.6	36.8	52.3
Other Gases	107.7	101.7	64.7	76.5
Total	2747.	2078.	1632.	2034.
TOTAL MATERIAL RECOVERY, WT. %	98.1	99.0	99.4	99.1

1 - Unreacted coal
2 - Oil, asphaltenes and preasphaltenes

APPENDIX C

MATERIAL BALANCES FOR TWO-STAGE THERMAL/CATALYTIC LIQUEFACTION TESTS

RUN NO.	7005	7006	7007	7008
COAL TESTED	ILLINOIS NO.6	DONKIN HR-7	LINGAN HR-6	PRINCE HB-7
CATALYST TESTED	KATALCO 550	KATALCO 550	AMOCAT 1 C	AMOCAT 1 C
MOISTURE, WT. %	3.03	1.65	1.32	3.30
ASH, WT. % DRY BASIS	11.16	5.02	4.43	9.10
HOURS ON STREAM	8.05	8.03	24.04	8.03
DURATION OF BALANCE PERIOD, HRS.	2.02	2.00	4.04	2.00
INPUT (g):				
H_2O	16.6	8.9	21.7	17.9
Ash	59.1	26.7	71.7	47.6
maf Coal	471.	504.	1547.	476.
Solvent	1478.	1460.	4436.	1463.
H_2	78.5	79.9	161.4	78.8
Total	2103.	2080.	6238.	2083.
OUTPUT (g):				
H_2O	70.4	42.5	110.2	88.8
Ash	47.2	18.4	63.8	38.1
Organic THF Insolubles[1]	19.7	32.8	131.0	31.5
THF Solubles[2]	1780.	1791.	5565.	1793.
H_2	46.6	49.3	90.7	40.0
Other Gases	80.6	107.0	210.1	93.0
Total	2045.	2041.	6171.	2084.
TOTAL MATERIAL RECOVERY, WT. %	97.2	98.1	98.9	100.1

1 - Unreacted coal
2 - Oil, asphaltenes and preasphaltenes

APPENDIX C

MATERIAL BALANCES FOR TWO-STAGE THERMAL/CATALYTIC LIQUEFACTION TESTS

RUN NO.	7009	7010	7011	7012
COAL TESTED	PRINCE HB-7	LINGAN HR-7	DONKIN HR-7	DONKIN HR-7
CATALYST TESTED	AMOCAT 1 C	AMOCAT 1 C	AMOCAT 1 C	AMOCAT 1 C
MOISTURE, WT. %	3.30	1.28	1.65	1.65
ASH, WT. % DRY BASIS	9.10	3.42	5.02	5.02
HOURS ON STREAM	9.48	8.02	8.02	10.03
DURATION OF BALANCE PERIOD, HRS.	2.00	2.03	1.99	2.03
INPUT (g):				
H_2O	17.3	7.3	9.0	9.2
Ash	46.1	19.3	26.9	27.5
maf Coal	460.	545.	509.	521.
Solvent	1415.	1546.	1475.	1507.
H_2	78.8	81.1	79.5	81.1
Total	2017.	2199.	2099.	2146.
OUTPUT (g):				
H_2O	77.9	38.1	46.7	39.6
Ash	35.6	16.3	21.3	21.7
Organic THF Insolubles[1]	30.2	32.6	31.3	35.0
THF Solubles[2]	1744.	1972.	1864.	1933.
H_2	44.0	49.5	47.6	54.4
Other Gases	82.5	90.7	90.7	78.4
Total	2014.	2200.	2102.	2162.
TOTAL MATERIAL RECOVERY, WT. %	99.9	100.0	100.1	100.8

1 - Unreacted coal
2 - Oil, asphaltenes and preasphaltenes

APPENDIX C

MATERIAL BALANCES FOR TWO-STAGE THERMAL/CATALYTIC LIQUEFACTION TESTS

RUN NO.	7014	7015	7016	7017
COAL TESTED	DONKIN HR-7	LINGAN HR-7	PRINCE HB-7	ILLINOIS NO. 6
CATALYST TESTED	AMOCAT 1 C	AMOCAT 1 C	AMOCAT 1 C	AMOCAT 1 C
MOISTURE, WT. %	1.65	1.28	3.30	3.03
ASH, WT. % DRY BASIS	5.02	3.42	9.10	11.16
HOURS ON STREAM	10.03	10.13	10.07	10.63
DURATION OF BALANCE PERIOD, HRS.	1.97	2.05	2.03	2.58
INPUT (g):				
H_2O	8.6	7.1	18.2	22.4
Ash	25.8	18.8	48.6	80.1
maf Coal	488.	531.	485.	637.
Solvent	1413.	1507.	1492.	2000.
H_2	130.8	136.2	132.6	168.3
Total	2066.	2200.	2176.	2908.
OUTPUT (g):				
H_2O	42.3	34.9	69.3	91.7
Ash	19.2	14.7	39.2	76.7
Organic THF Insolubles[1]	29.7	31.2	32.8	31.5
THF Solubles[2]	1795.	1909.	1820.	2476.
H_2	90.3	101.2	98.0	112.9
Other Gases	102.2	106.2	95.0	116.7
Total	2079.	2197.	2154.	2906.
TOTAL MATERIAL RECOVERY, WT. %	100.6	99.9	99.0	99.9

1 - Unreacted coal
2 - Oil, asphaltenes and preasphaltenes

APPENDIX C

MATERIAL BALANCES FOR TWO-STAGE THERMAL/CATALYTIC LIQUEFACTION TESTS

RUN NO.	7018	7019
COAL TESTED	LINGAN HR-7	PRINCE HB-7
CATALYST TESTED	AMOCAT 1 C	AMOCAT 1 C
MOISTURE, WT. %	1.28	3.30
ASH, WT. % DRY BASIS	3.42	9.10
HOURS ON STREAM	10.07	10.12
DURATION OF BALANCE PERIOD, HRS.	1.98	4.02
INPUT (g):		
H_2O	7.0	41.0
Ash	18.4	109.3
maf Coal	519.	1092.
Solvent	1471.	2898.
H_2	131.5	286.5
Total	2147.	4427.
OUTPUT (g):		
H_2O	38.3	153.6
Ash	13.8	91.1
Organic THF Insolubles[1]	28.7	76.5
THF Solubles[2]	1798.	3646.
H_2	91.9	214.2
Other Gases	131.0	189.4
Total	2102.	4371.
TOTAL MATERIAL RECOVERY, WT. %	97.9	98.7

1 - Unreacted coal
2 - Oil, asphaltenes and preasphaltenes

APPENDIX C

MATERIAL BALANCES FOR RCL-7020

OPERATING PERIOD, HRS.	0-12	12-24	24-36	36-48
COAL TESTED	PRINCE HB-7	PRINCE HB-7	PRINCE HB-7	PRINCE HB-7
CATALYST TESTED	AMOCAT 1C	AMOCAT 1C	AMOCAT 1C	AMOCAT 1C
MOISTURE, WT. %	3.07	3.07	3.07	3.07
ASH, WT. % DRY BASIS	8.95	8.95	8.95	8.95
INPUT (g):				
H_2O	110.7	106.8	108.4	108.0
Ash	312.9	301.7	306.3	305.2
maf Coal	3184.	3069.	3116.	3105.
Solvent	9276.	8943.	9080.	9046.
H_2	664.2	664.2	664.2	664.2
Total	13548.	13085.	13275.	13228.
OUTPUT (g):				
H_2O	477.6	449.6	452.7	460.3
Ash	235.3	266.5	303.7	363.7
Organic THF Insolubles[1]	225.2	225.4	251.3	231.8
THF Solubles[2]	11799.	11070.	11097.	11070.
H_2	481.9	475.9	475.5	462.3
Other Gases	563.1	534.5	575.7	558.0
Total	13782.	13022.	13156.	13146.
TOTAL MATERIAL RECOVERY, WT. %	101.7	99.5	99.1	99.4

1 - Unreacted coal
2 - Oil, asphaltenes and preasphaltenes

APPENDIX C

MATERIAL BALANCES FOR RCL-7020

OPERATING PERIOD, HRS.	48-60	60-72	72-84	84-96
COAL TESTED	PRINCE HB-7	PRINCE HB-7	PRINCE HB-7	PRINCE HB-7
CATALYST TESTED	AMOCAT 1C	AMOCAT 1C	AMOCAT 1C	AMOCAT 1C
MOISTURE, WT. %	3.07	3.07	3.07	3.07
ASH, WT. % DRY BASIS	8.95	8.95	8.95	8.95
INPUT (g):				
H_2O	106.6	106.5	104.7	105.6
Ash	301.2	300.9	295.9	298.5
maf Coal	3064.	3061.	3010.	3037.
Solvent	8929.	8918.	8771.	8848.
H_2	664.2	664.2	664.2	664.2
Total	13065.	13051.	12846.	12953.
OUTPUT (g):				
H_2O	442.2	438.4	445.9	444.8
Ash	244.0	284.0	283.7	284.5
Organic THF Insolubles[1]	255.4	250.3	259.2	252.8
THF Solubles[2]	11089.	10940.	10698.	10841.
H_2	442.4	456.5	455.7	455.8
Other Gases	580.6	560.7	607.4	610.2
Total	13054.	12930.	12750.	12889.
TOTAL MATERIAL RECOVERY, WT. %	99.9	99.1	99.3	99.5

1 - Unreacted coal
2 - Oil, asphaltenes and preasphaltenes

APPENDIX C

MATERIAL BALANCES FOR RCL-7020

OPERATING PERIOD, HRS.	96-108	108-120	120-132	132-144
COAL TESTED	PRINCE HB-7	PRINCE HB-7	PRINCE HB-7	PRINCE HB-7
CATALYST TESTED	AMOCAT 1C	AMOCAT 1C	AMOCAT 1C	AMOCAT 1C
MOISTURE, WT. %	3.07	3.07	3.07	3.07
ASH, WT. % DRY BASIS	8.95	8.95	8.95	8.95
INPUT (g):				
H_2O	103.5	105.0	104.8	106.9
Ash	292.3	296.8	296.1	302.1
maf Coal	2974.	3020.	3012.	3073.
Solvent	8665.	8798.	8777.	8953.
H_2	664.2	664.2	664.2	664.2
Total	12699.	12884.	12854.	13099.
OUTPUT (g):				
H_2O	434.6	443.2	436.8	416.0
Ash	298.1	307.3	290.7	292.4
Organic THF Insolubles[1]	255.2	275.4	246.4	267.3
THF Solubles[2]	10670.	11076.	10429.	10918.
H_2	460.0	450.3	439.8	443.4
Other Gases	595.3	680.0	691.7	675.7
Total	12713.	13232.	12534.	13013.
TOTAL MATERIAL RECOVERY, WT. %	100.1	102.7	97.5	99.3

1 - Unreacted coal
2 - Oil, asphaltenes and preasphaltenes

APPENDIX D

RESULTS OF HPLC APPLIED TO COAL-DERIVED LIQUIDS

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RESULTS OF HPLC APPLIED TO COAL-DERIVED LIQUIDS

During Phase VII, high performance liquid chromatography (HPLC) was used to separate coal liquids into saturates, aromatics and polar compounds. Details pertaining to the HPLC method have been given in an earlier report (6).

In the HPLC method, separation of coal liquids into "double bond" fractions was achieved. A number of model compounds were used to define the specific regions of the chromatograph as shown in Figure 11. The regions were defined by the number of double bonds in a given hydrocarbon molecule ranging from 0 (saturates) to 8 (tetracyclic PAH). Elution profiles for hydrogenated anthracene oil and the pentane soluble fraction of C_5^+ liquids from selected two-stage coal liquefaction experiments are shown in Figures 12 - 17. The C_5^+ liquids were obtained by combining separator overheads light oil and separator bottoms liquid recovered by vacuum filtration at 75°C.

Results of HPLC analysis presented in Table 15 show that pentane soluble oil from Run Nos. 7014, 7015, 7016 and 7017 contains more saturates and less aromatics than the reaction solvent, HAO 71. Furthermore, the product oils contain a higher level of polar compounds than HAO 71.

Hydrocarbon group analysis of the pentane soluble fraction of C_5^+ liquid product from RCL-7020 is shown in Table 16. The product oil contained 26.73% saturates, 61.26% aromatics and 12.01% polar compounds. Concentrations of 5, 6, 7 and 8 "double bond" aromatics were considerably lower in the product oil than in the start-up solvent.

Figure 11 - HPLC ELUTION PROFILES FOR MODEL COMPOUNDS

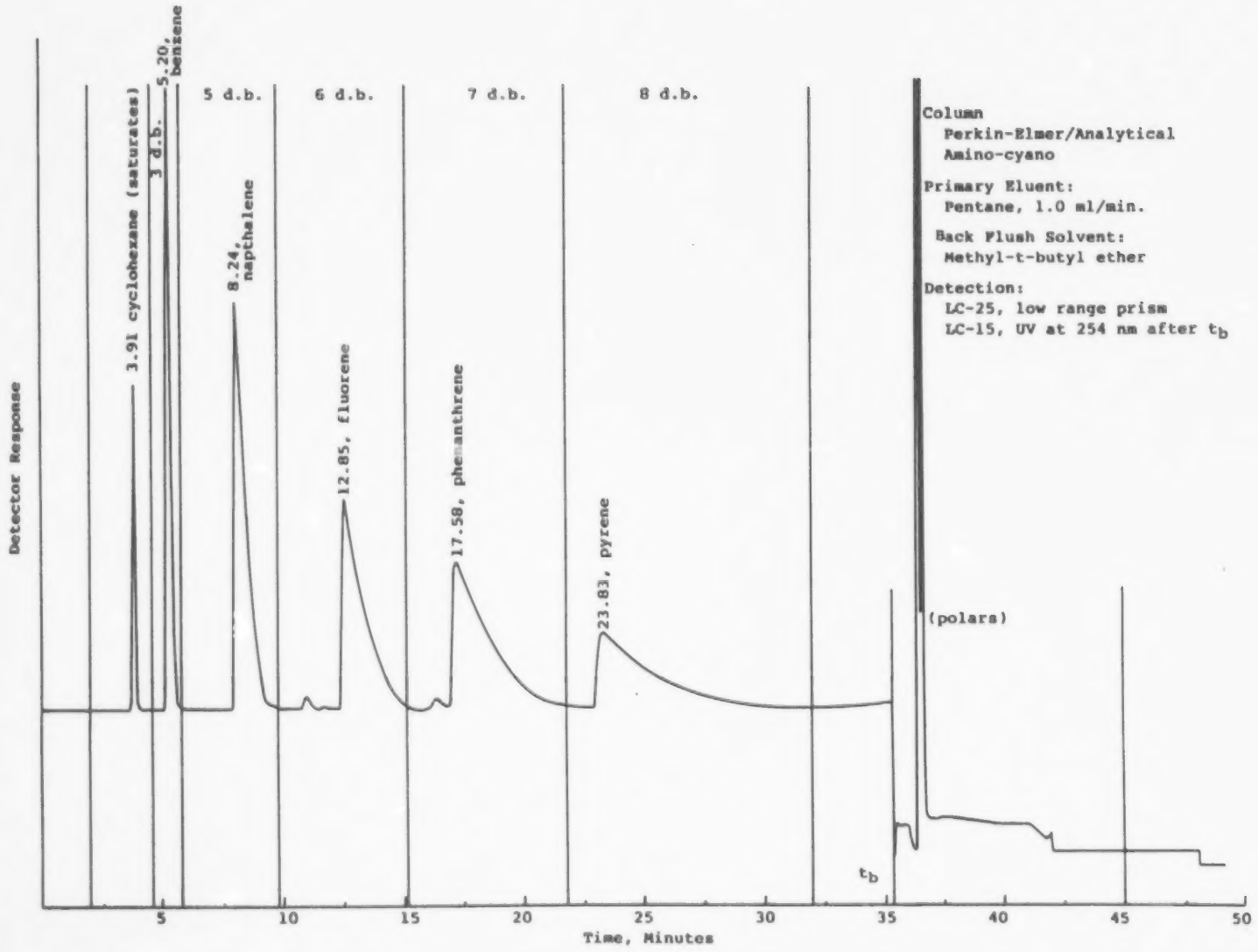
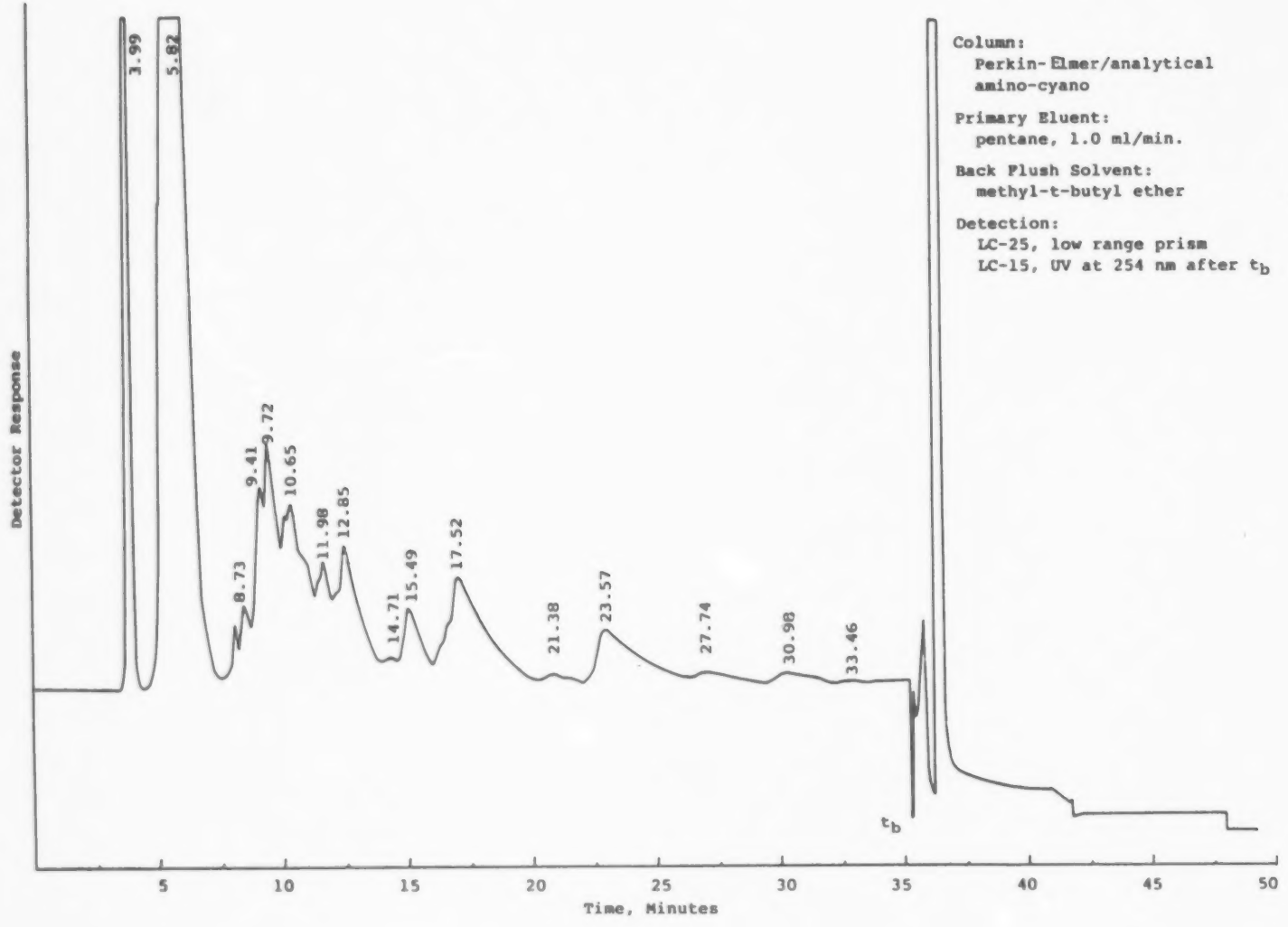


Figure 12 - HPLC SEPARATION OF HYDROGENATED ANTHRACENE OIL WITH 9.01 WT.% HYDROGEN



Fgiure 13 - HPLC SEPARATION OF PENTANE SOLUBLE FRACTION OF C_5^+ LIQUID FROM RUN NO. 7014

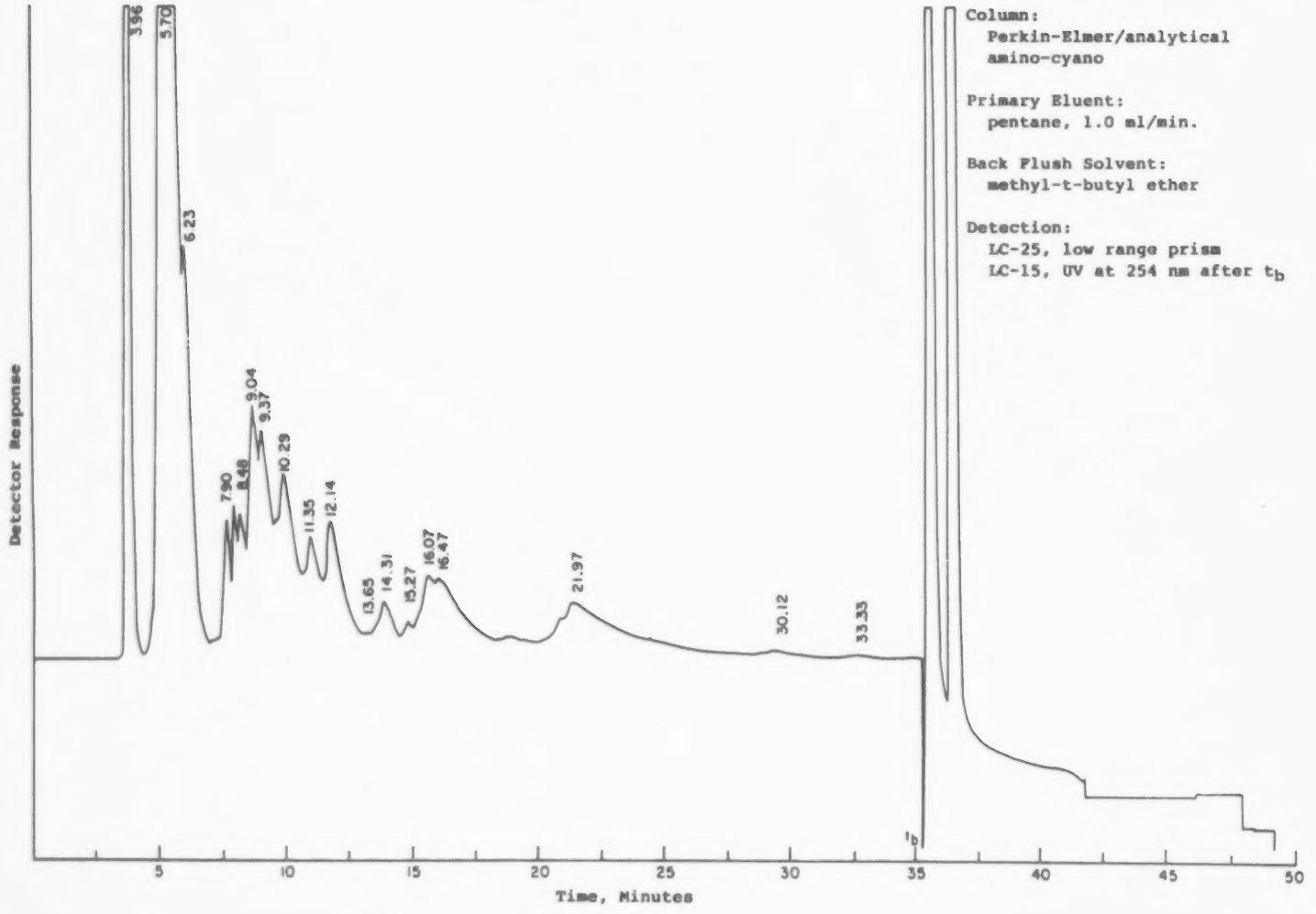


Figure 14 - HPLC SEPARATION OF PENTANE SOLUBLE FRACTION OF C_5^+ LIQUID FROM RUN NO. 7015

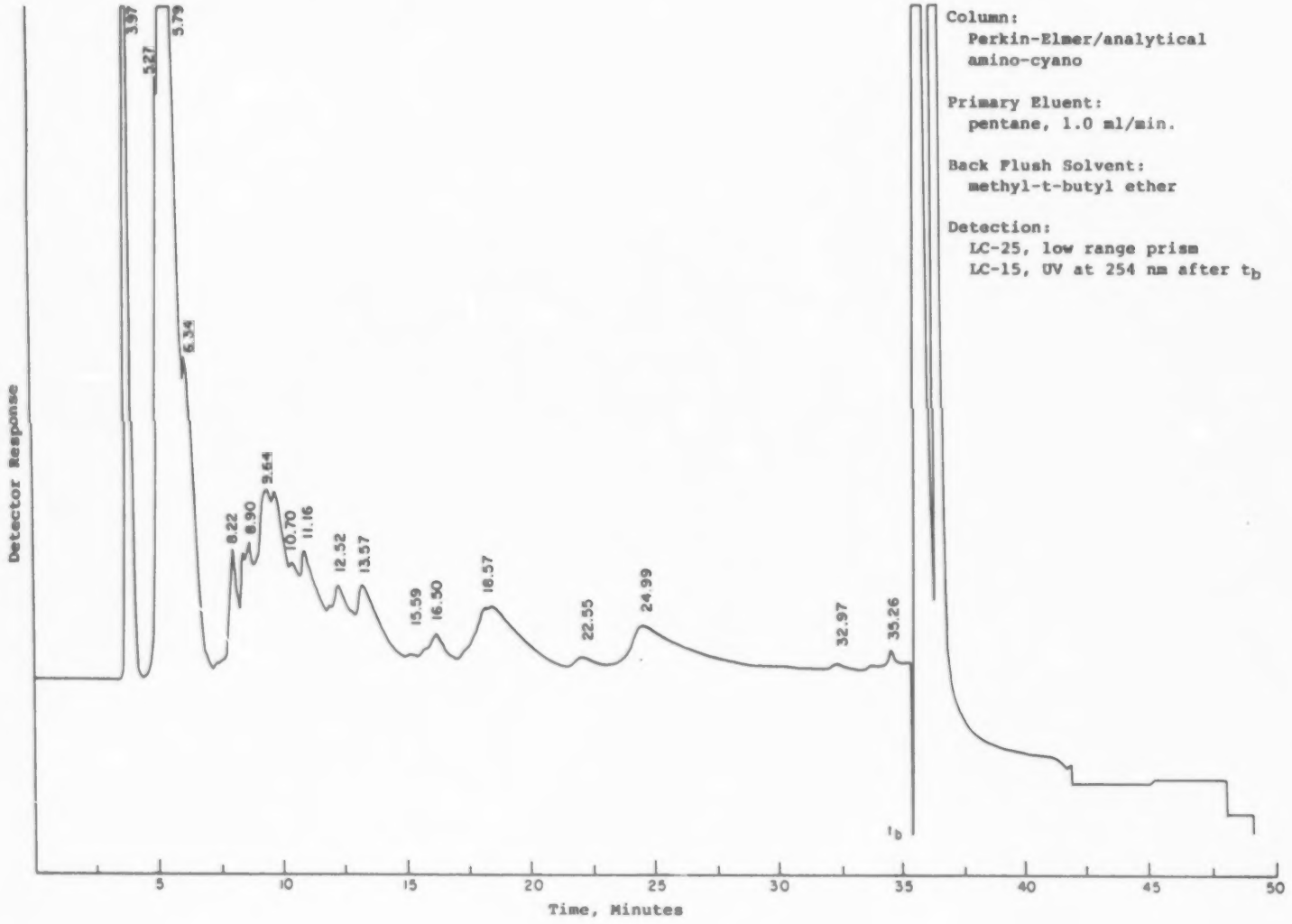


Figure 15 - HPLC SEPARATION OF PENTANE SOLUBLE FRACTION OF C_5^+ LIQUID FROM RUN NO. 7016

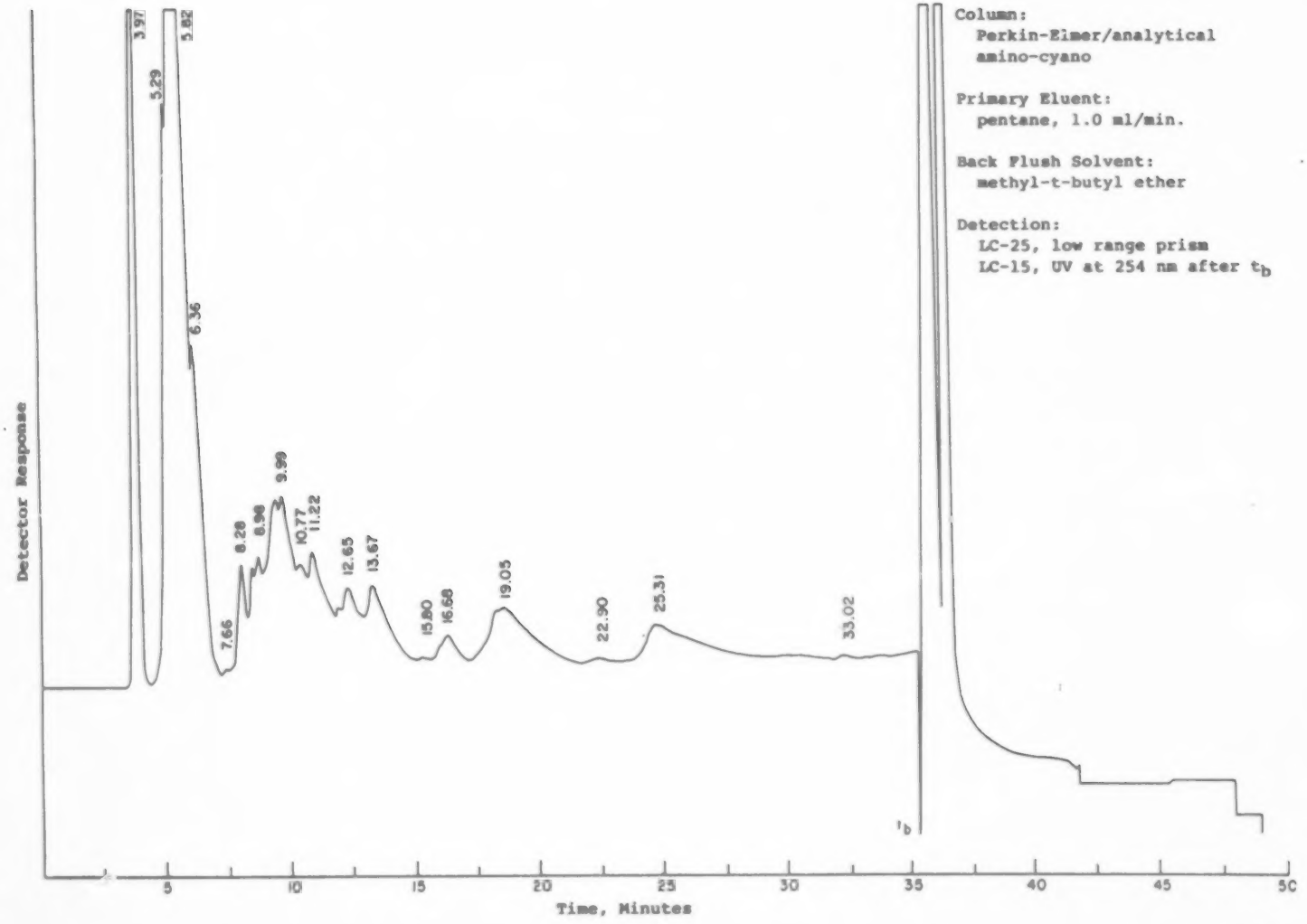


Figure 16 - HPLC SEPARATION OF PENTANE SOLUBLE FRACTION OF C_5^+ LIQUID FROM RUN NO. 7017

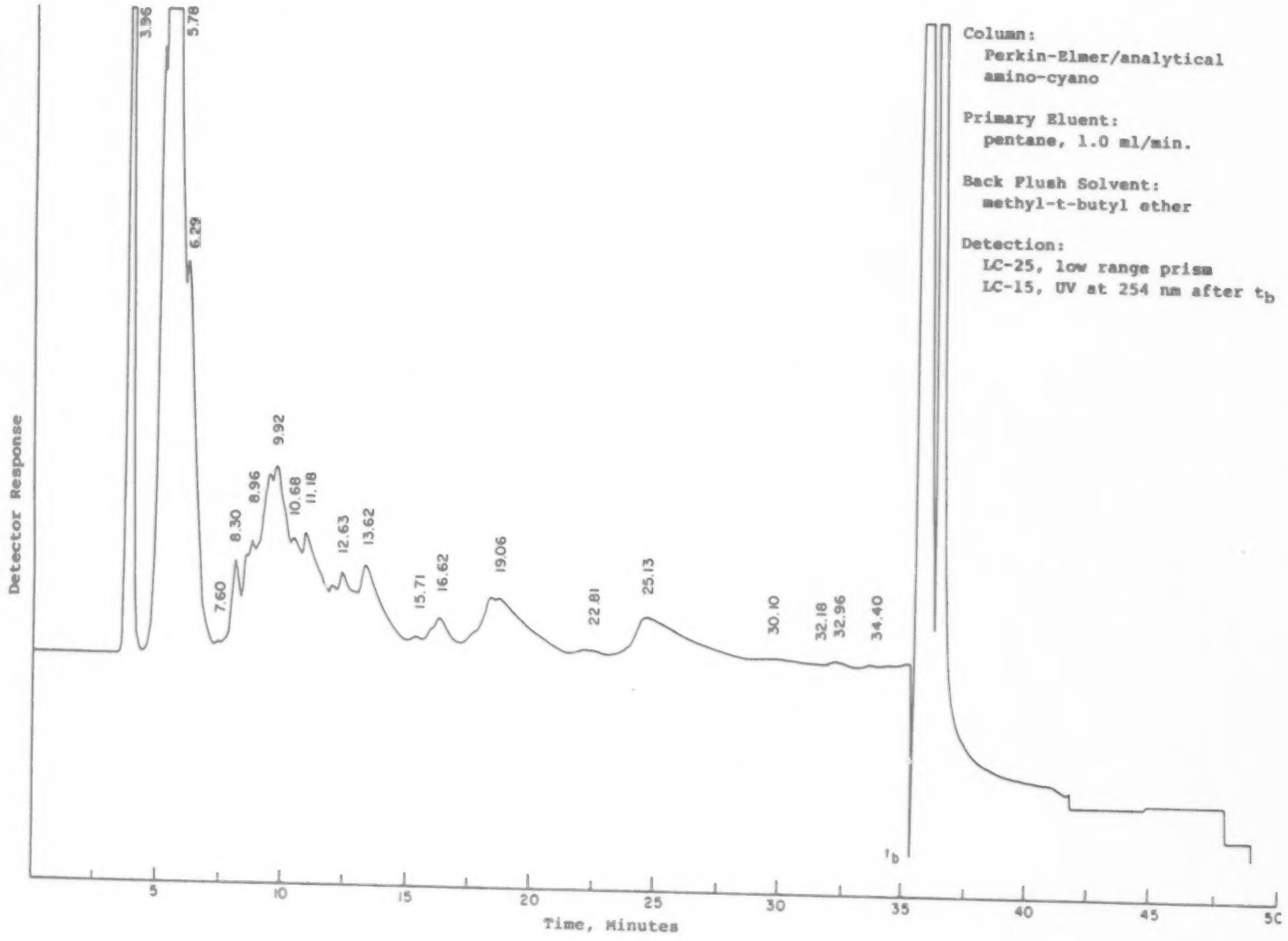


Figure 17 - HPLC SEPARATION OF PENTANE SOLUBLE FRACTION OF C_5^+ LIQUID FROM RCL-7020

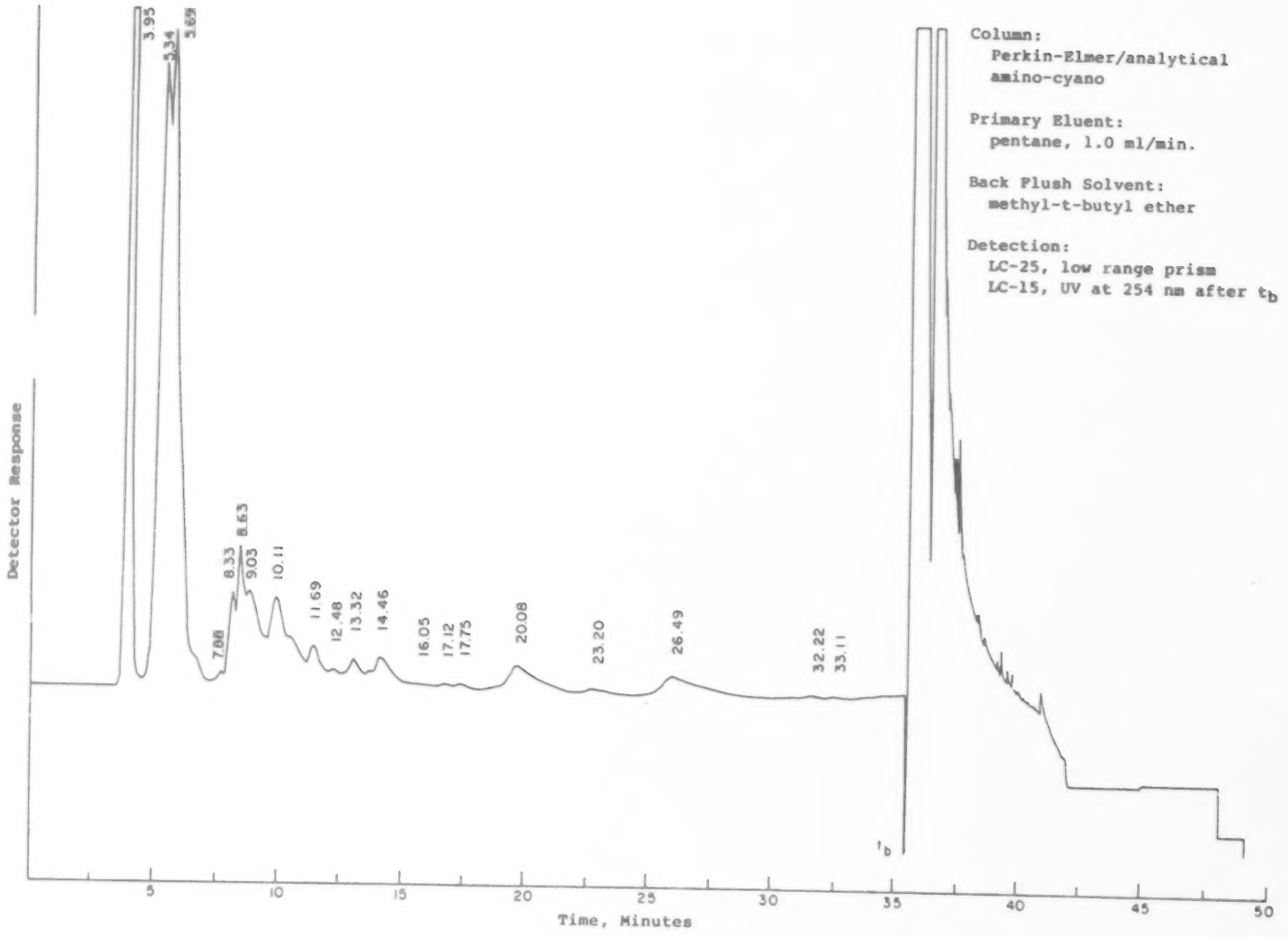


TABLE 15

HPLC SEPARATION OF HYDROGENATED ANTHRACENE OIL AND THE PENTANE SOLUBLE FRACTION OF C_5^+ LIQUIDS FROM SELECTED COAL LIQUEFACTION EXPERIMENTS

SAMPLE DESCRIPTION	HAO 71	RUN NO. 7015 LINGAN HR-7	RUN NO. 7014 DONKIN HR-7	RUN NO. 7016 PRINCE HB-7	RUN NO. 7017 ILLINOIS NO. 6
Specific Gravity, 60/60°F	1.0262	1.0215	1.0125	1.0172	1.0108
Carbon, Wt. %	89.73	89.53	89.86	90.12	90.09
Hydrogen, Wt. %	9.01	8.89	8.83	9.04	9.15
Nitrogen, Wt. %	0.10	0.19	0.24	0.18	0.18
Sulphur, Wt. %	0.005	0.038	0.016	0.029	0.051
Oxygen, Wt. %	0.35	0.47	0.44	0.49	0.46
Pentane Insolubles, Wt. %	NIL	4.79	3.37	2.70	1.71
HPLC DATA FOR PENTANE SOLUBLE FRACTION:					
Saturates, Wt. %	13.18	15.25	16.02	14.65	14.90
Aromatics, Wt. %	82.77	71.40	73.14	71.50	73.24
3 double bonds	11.82	11.68	23.03	12.21	13.90
5 double bonds	28.72	24.68	22.35	24.66	26.79
6 double bonds	23.99	16.68	11.52	15.17	16.95
7 double bonds	8.44	8.35	8.47	8.58	6.79
8 double bonds	9.80	10.01	7.77	10.88	8.81
Polars, Wt. %	4.05	13.35	10.84	13.85	11.86

TABLE 16

HPLC SEPARATION OF PENTANE SOLUBLE FRACTION
OF C_5^+ LIQUID PRODUCT FROM RCL-7020

Specific Gravity, 60/60°F	0.8976
Carbon, Wt. %	89.29
Hydrogen, Wt. %	10.76
Nitrogen, Wt. %	0.24
Sulphur, Wt. %	0.071
Oxygen, Wt. %	0.78
Pentane Insolubles, Wt. %	3.01
HPLC DATA FOR PENTANE SOLUBLE FRACTION:	
Saturates, Wt. %	26.73
Aromatics, Wt. %	61.26
3 double bonds	12.38
5 double bonds	22.24
6 double bonds	13.06
7 double bonds	6.35
8 double bonds	7.23
Polars, Wt. %	12.01

APPENDIX E

OPERATING PROCEDURE FOR

TWO-STAGE CONTINUOUS COAL LIQUEFACTION UNIT

PREPARATION OF UNIT PRIOR TO ACTUAL OPERATION

This procedure is normally carried out the day before a run.

1. Open valves 1, 7, 11, 14, 15, 116, 119 and 122.

2. Depressure the system to 345 kPa by opening valves 17 and 18. Close valves 17 and 18.

3. Open valve 2 and valve 8 and pressurize the system as well as the 7.6 liter emergency blowdown vessel to 4.14 MPa. Close valve 8 and continue pressurizing the system until the pressure is at the desired level either 10.34 MPa or 13.79 MPa. Close valve 2.

4. Leave for a few hours and monitor pressure.

5. Set the forward pressure regulator and the back pressure regulator to the desired settings.

6. Turn on the cooling water to the magnedrive stirrers.

7. Set the controllers at the desired settings for both the first-stage and second-stage reactors.

	Stage 1	Stage 2
reactor controller °C	343	288
furnace controller °C	343	316
zone controllers, %	60	100

8. Set the automatic timer to activate the reactor furnace temperature controllers according to temperature programming schedule. Turn the reactor temperature controller switches to the "ON" position and ensure they are not activated.

9. Set the temperature controller for high pressure separator No. 1, V-5, at 300°C.

10. Set the Automatic timer to activate V-5 temperature controller at 4 hours prior to anticipated start-up. Turn the controller switch to the "ON" position and ensure it is not activated.

11. Ensure that alarm switch No. 14 is deactivated.

ACTUAL OPERATION

Start-Up

1. Gradually increase the furnace controllers for stage 1 and stage 2 to the desired levels. NEVER allow the heating rate of the reactors to exceed 120°C per hour.

2. Turn on heating tapes on slurry transfer lines:

   No. 1 - 33%, at start of run gradually increase to 100%

   No. 2 - 33%, at start of run gradually increase to 66%

   No. 3 - 33%

   No. 4 - 33%

3. Activate heater on high pressure separator No. 2, V-7.

4. Turn on condenser cooling water.

5. Activate K-Ray electronics for liquid level measurement system.

6. After one hour open the window on the radioactive source.

7. Turn on the hydrogen flow control system fifteen minutes prior to run.

Procedure

   a) Turn on the power.
   b) Open valve 3.
   c) Set the air pressure at 138 kPa.
   d) Adjust the setpoint knob until the ball is in the center, then switch from manual to automatic.
   e) Set the pen to the desired value.
   f) Open valve 123 and close valve 122.
   g) Allow the hydrogen flow control system to stabilize.
   h) Monitor exit gas flow rate for 15 minutes.

8. Purge the feed tank with nitrogen 15 minutes prior to slurry addition. Maintain a small nitrogen flow throughout run.

9. Charge V-1 with the slurry. Open valve 101 and turn on the circulating pump.

10. Turn off the circulating pump for 15 seconds and then turn circulating pump back on. This fills void spaces or gas pockets in the pump with slurry and ensures proper weight readings on the toledo scale. Turn on the alarm switch No. 14 in the side panel.

11. When the temperatures of the reactors are at the desired levels open valve 102 and purge the high pressure metering pump with slurry until the liquid is gas free.

Run

12. Open the plant valve (valve 5 or 6) and activate the high pressure metering pump.

13. Turn on the V-3, V-4 and V-5 magnedrive stirrers.

14. Turn on the cooling fan for magnedrive stirrer on V-4.

15. Monitor the pressures, temperatures, slurry feedrate and vent gas flow rates throughout the run.

16. A rapid increase in the reactor 1 temperature will occur 10 to 20 minutes after the high pressure metering pump has been activated. When the reactor temperature reaches 4°C below the target temperature turn the furnace temperature controller down until the red light goes off. Increase the furnace temperature controller back to the original setpoint when reactor 1 temperature levels off.

17. Open valves 111, 107, 104 and 105 and purge V-9 with nitrogen, 10 minutes prior to step 18.

18. After 2.3 kg of slurry has been pumped, open valve 22.

19. Set timer K-5 for the automatic letdown (ALD) system to maintain a constant level in V-5.

20. Open valve 19.

21. Set the selector switch on "Automatic During High Alert" and activate the automatic letdown system.

22. After the first ALD cycle (mark this on data sheet) close valve 105.

23. Sample vent gas streams 15 minutes prior to steady state sampling period.

24. Record the toledo scale weight and deactivate the circulating pump for 15 seconds 15 minutes prior to steady state sampling period. Reactivate the circulating pump and record the toledo scale weight.

25. Drain liquid from the separator overheads knock-out (V-7) periodically and 5 minutes prior to steady state sampling period by opening and closing valves 17 and 18 sequentially.

26. To initiate the steady state sampling period, record the toledo scale weight after valve "A" has closed on the final ALD cycle of the slop period.

27. Record the wet test meter reading and the circulating pump pan weight.

28. Open valves 108 and 23. Close valves 22 and 107. Open valve 106 and start a nitrogen purge.

29. On the next ALD cycle, the first in the steady state sampling period, mark the stroke made on the hydrogen flow recorder.

30. Close valve 106.

31. Open valve 120 and drain V-15. Close valve 120.

32. Sample vent gas streams 15 minutes prior to end of steady state sampling period.

33. Five minutes prior to end of run, drain liquid from separator overheads knock-out, V-7, by opening and closing valves 17 and 18 sequentially.

34. At the end of the period, record the toledo scale weight after valve "A" has closed on the final ALD cycle of the sampling period. Close valve 19 and deactivate the automatic letdown system.

35. Record the wet test meter reading and the circulating pump pan weight. Empty contents of V-15.

36. Turn the reactor controller setpoints on stage 1 and stage 2 reactors down to 260°C. Turn the furnace controller setpoints on stage 1 and stage 2 reactors down to 204°C. Reduce voltage to heating tapes to 33% on slurry transfer lines. Turn off high pressure separator heaters.

37. Record the toledo scale weight and deactivate the circulating pump for 15 seconds. Reactivate the circulating pump and record the toledo scale weight.

38. Shut off the hydrogen flow control system.

Procedure

a) Turn the regulator knob until the ball is in the center.
b) Switch from automatic to manual
c) Shut off the air flow.
d) Close valve 3.
e) Turn off the power.

39. Deactivate the high pressure metering pump.

40. Close the plant valve (valve 5 or 6) and valves 11 and 14.

41. Deactivate the circulating pump and drain slurry from the feed tank.

42. Close valve 101 and 102 and add flush oil to V-1, open valve 101 and turn on the circulating pump.

43. Open valve 109. Manually withdraw slurry product from V-5 by opening valves 25 and then 24. Close valves 24 and then 25. Close valve 109. Open valve 13 and repressure V-5 to 6.89 MPa and then repeat step 43.

44. Open valve 120 and drain contents of V-15. Close valve 120.

45. Open valves 14 and 15 to equalize the pressure in V-5, V-6 and V-7.

46. Open valve 11 to equalize the pressure throughout the system.

47. Open valve 17. Slowly open valve 18 to release system pressure to ca. 13.79 MPa. Close valve 17 and then valve 18.

48. Close valve 11.

49. Close the interstage valve (valve 7).

50. Transfer contents of V-3 into the 7.6 liter high pressure vessel (which has been pressurized to 4.14 MPa hydrogen) through valve 8. Leave valve 8 open for 5 minutes and then close valve 8.

51. Transfer contents of V-4 into the 7.6 liter pressure vessel through valve 9. Leave valve 9 open for 5 minutes and then close valve 9.

52. Open valve 11 and equalize pressure between V-4, V-5 and V-6.

53. Open valve 2 and add high pressure hydrogen to V-3 (stage 1 reactor) until its pressure is 689 kPa in excess of the stage 2 reactor pressure. Close valve 2.

54. Open the interstage valve (valve 7) to equalize system pressure.

55. Open valve 122 and close valve 123.

Flush Cycle and Shutdown

56. Reactivate the reactor controller setpoints in both reactors to 316°C. Reactivate the furnace controller setpoints in both reactors to 427°C. Turn on the heater on separator No. 1 to 300°C. Increase voltage on line heaters to 66% for the preheater and 33% for the interstage and slurry transfer lines.

57. Open valve 102 and purge the high pressure metering pump with oil until the liquid is gas free. Open the plant valve and activate the high pressure metering pump.

58. Note: Keep temperatures of stage 1 and stage 2 reactors between 288°C and 316°C during flush cycle.

59. Open valve 111 and 120 and drain V-15. Close valve 111.

60. When approximately 3.0 kg of oil has been pumped into V-5, open valves 111, 108, 23 and 19. Activate the ALD system allowing it to operate until at least 6 kg of flush oil has been pumped. Close valve 19, allow one more cycle. Deactivate the ALD system and close valves 23, 108 and 111.

61. Pull plug out for electronics on ALD system and close window on radioactive source.

62. Deactivate the metering pump.

63. Shut off all heating tapes, heaters and the reactor furnaces.

64. Turn off the condenser cooling water. Leave magnedrive water on overnight.

65. Close the plant valve and valves 11 and 14.

66. Withdraw oil from V-5.

67. Open valves 14 and 15 to equalize the pressure in V-5, V-6 and V-7.

68. Open valve 11 to equalize the pressure throughout the system.

69. Close the interstage valve and valve 11.

70. Transfer contents of V-3 into the 7.6 liter high pressure vessel by opening valve 8. Let drain for 5 minutes. Prior to closing valve 8, open valve 2 and slowly add pressure to V-3 and the 7.6 liter vessel until the pressure is 4.83 MPa, then close valve 2 and valve 8.

71. Transfer contents of V-4 into 7.6 liter high pressure vessel by opening valve 9. Let stand for 5 minutes then close valve 9.

72. Open valve 11 and equalize pressure in V-4, V-5 and V-6.

73. Follow steps 53 and 54. Then open valve 2 and add hydrogen to the system until the pressure is 11.38 MPa.

74. Drain flush oil from V-1.

75. Turn off electronics for toledo scale and digital pressure meter.

76. Shut off alarm switch No. 14 in side panel.

POST RUN OPERATIONS

This procedure is normally carried out the day after the run.

1. Open valve 2 and pressurize the system to 10.34 MPa.

2. Close the interstage valve (valve No. 7) and valves 11 and 14.

3. Depressure the 7.6 liter pressure vessel to 4.83 MPa.

4. Drain contents of V-5 by opening valves 110, 26 and 24. Close valves 24, 26 and 110.

5. Open valve 14 and equalize the pressure in V-5, V-6 and V-7.

6. Empty contents of V-3 by opening valve 8. Close valve 8.

7. Depressure the 7.6 liter pressure vessel to 4.83 MPa.

8. Empty contents of V-4 by opening valve 9. Close valve 9.

9. Open valve 11.

10. Follow steps 53 and 54. Then open valve 2 and add hydrogen to the system until the pressure is ca. 13.79 MPa.

11. Depressure the 7.6 liter pressure vessel to 1.38 MPa. Empty contents of the 7.6 liter vessel into the 10 liter low pressure vessel.

12. Follow steps 1 to 4 on page E-2.

SCHEMATIC DIAGRAM OF TWO-STAGE COAL LIQUEFACTION BENCH UNIT